ARMY RESEARCH LABORATORY

Fourier Transform Photoacoustic Infrared Spectroscopy of Propellant Formulations, Propellant Ingredients, and Energetic Materials

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Kevin L. McNesby

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13. ABSTRACT ***(Maximum 200 words)***

Infrared spectra of 48 energetic materials, propellant ingredients, and propellant formulations have been measured using a Fourier transform infrared (FTIR) spectrometer employing photoacoustic (PA) detection. Spectra are divided among the categories of propellant formulations, neat energetic materials, binders, plasticizers, stabilizers and others. Spectra of propellant formulations have been annotated, where appropriate, to indicate features due to individual ingredients.

14. SUBJECT TERMS

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PREFACE

This work was performed while the author held a National Research Council-Army Research Laboratory Research Associateship.

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1. INTRODUCTION

Propellant formulations and their ingredients are often opaque and/or poorly reflective, making it difficult to measure infrared (IR) spectra using conventional techniques. Techniques for measuring IR spectra, which rely on detection of beam attenuation, such as transmission, reflectance, and attenuated total reflectance (ATR), are often not suitable for such samples or require elaborate sample preparation. To avoid saturation, transmission spectroscopy in the mid-IR spectral region requires that the path length through a typical propellant sample be on the order of 5–10 μm. Reflectance spectroscopy of many crystalline energetic materials may be sensitive to sample alignment and frequently requires mathematical corrections to the raw data. ATR spectroscopy requires that the sample be brought into intimate contact with an internal reflection element and may not be suitable for many solid samples. Fourier transform infrared (FTIR) spectroscopy employing photoacoustic (PA) detection may be used to overcome many of the limitations posed by these traditional techniques. PA detection requires minimal sample preparation and may be successfully used with opaque, poorly reflecting samples and samples with widely varying morphology (McClelland et al. 1993). Additionally, for some samples, the PA technique allows measurement of vibrational spectra at discrete depths beneath the sample surface (Palmer 1993).

This report contains a PA IR spectral library of propellant formulations, neat ingredients, and energetic materials. At the present time, we know of no commercially available IR libraries dealing specifically with spectra of propellant formulations, propellant ingredients, and energetic materials. We believe such a library would have applications in and beyond those of the propellant community, especially in the areas of environmental site remediation, identification of unknowns, and the analysis of stored propellants that may be in varying states of decomposition.

2. BACKGROUND

Following the excitation of vibrational modes by the absorption of IR radiation, relaxation processes dissipate the absorbed energy. Most of the energy is distributed among the normal modes according to the Boltzmann distribution, resulting in a temperature rise of the sample. If the intensity of the incident IR radiation is constant, the rate of heat generation in the sample will be constant. If, however, the intensity of the incident IR radiation is modulated, the heating of the sample will be modulated. The modulation of the sample surface temperature causes the gas adjacent to the sample surface to expand at the modulation frequency of the incident radiation. The modulated expansion of the gas adjacent to the

sample causes pressure pulses in the gas. When the modulation frequency is in the acoustic range, the pressure pulses may be detected using a microphone. The magnitude of the signal reaching the microphone, during one modulation period, is proportional to the strength of the sample absorption. When the modulation of the IR radiation is accomplished using an interferometer, a Fourier transform of the microphone output yields the IR spectrum of the sample (Dittmar, Palmer, and Carter 1994).

PA signal generation is dependent on the transfer of vibrational energy from the sample to translational energy of the gas adjacent to the sample surface. This results in a time lag (phase shift) between IR absorption by the sample and detection of signal by the microphone. The magnitude of the time lag is dependent on the depth within the sample at which absorption occurs. This time lag between when the IR radiation is absorbed and when the resulting sound is detected by the microphone is manifested as a phase difference between IR source modulation frequency and microphone output frequency. The significance of the phase difference is that it can be used to calculate the depth beneath the sample surface of the absorbing species (Palmer 1993). At high modulation frequencies, only heat produced by absorption on or near the surface contributes to the signal detected during the modulation period. At lower modulation frequencies, the detected signal contains contributions from absorption deeper within the sample. Comparison of spectra measured at different modulations frequencies can reveal concentration profiles and/or stratification of absorbing species in the sample surface region (Dittmar 1994).

IR spectral depth profiling by PA detection is most easily accomplished using a step-scan interferometer employing phase-sensitive lock-in detection. This is because in the step-scan mode of operation, the modulation of the IR intensity is not wavelength-dependent. The technique involves positioning the moving mirror of the interferometer at a known retardation (optical path difference) and modulating the throughput of the interferometer at this mirror position by either mechanically chopping the beam or by applying a small amplitude sinusoidal "dither" to the moving mirror. The frequency of the modulation (the chopping or dithering frequency) at the nominally fixed-mirror position is then used as the reference frequency by the lock-in amplifier. In rapid scan interferometry, the IR modulation frequency and, thus, the effective sampling depth at which absorption can be detected is wavelength-dependent. The ability to preserve spectral multiplexing while removing the optical retardation dependence of the wavelength modulation, achievable by step-scan operation (Palmer 1993), allows uniform depth profiling over the full spectral range of the instrument. By varying the phase at which signal is collected relative to the chopping or dithering frequency, a single spectrum may be employed to yield absorption spectra at several discrete depth intervals beneath the sample surface (Palmer 1993).

3. EXPERIMENTAL

All the spectra in this library were measured using an MTEC 100 PA accessory and a Mattson Polaris spectrometer operating at a spectral resolution of 4 cm^{-1}. The spectra are the result of 32 or 64 coadded scans of the interferometer and have been ratioed to a spectrum of carbon black powder measured on the same day. Ratioing the spectra to the spectrum of carbon black normalizes the spectra to the instrumental throughput and the detector response (Palmer 1993). This is equivalent to ratioing a transmission spectrum to the spectrum of the open beam. Samples were placed in the sample holder of the MTEC accessory (a 6-mm-diameter cylindrical cup approximately 3 mm in depth), and the accessory was purged with dry helium gas to remove water vapor and CO_2 gas. An additional benefit of purging with helium is that the low heat capacity of the gas gives a signal intensity two to three times that of a cell filled with dry air.

Samples of propellant formulations, in the form of thin cross sections of grains and cords, were prepared by cutting with a razor blade. Samples of propellant formulations, when in the form of sheets, were prepared using a small sheet-metal punch. Samples of neat ingredients and energetic materials, when in powder form, were added to the sample cup using a small spatula and metal funnel. Samples of neat ingredients and energetic materials, when in liquid form, were added to the sample cup with a disposable pipet.

4. DISCUSSION AND RESULTS

The spectral library is divided into six categories. These categories are propellant formulations (Appendix A), energetic materials (Appendix B), binders (Appendix C), plasticizers (Appendix D), stabilizers (Appendix E), and others (Appendix F). The category of propellant formulations has been further broken down into four subcategories based on the formulation type. These formulation types are single-base propellants (nitrocellulose-based), double-base propellants (nitrocellulose- and nitroglycerin-based), triple-base propellants (nitrocellulose-, nitroglycerin-, and nitroguanidine-based), and nitramine-based (usually RDX- or HMX-based, or a combination of double base and nitramine-based). A list of the propellant formulations for which photoacoustic Fourier-transform infrared (PA-FTIR) spectra are reported here, arranged by propellant type, is given in Table 1. Table 2 lists the neat ingredients for which spectra are reported. Table 3 shows the major components of the propellant formulations listed in Table 1. Note that the List of Abbreviations section at the end of the report provides all the chemical abbreviations along with their spelled-out versions.

Table 1. Propellant Formulations

Single Base	Double Base	Triple Base	Nitramine-Based
M10	JA2 RPD351 M44, M9	M30	C4, XM39, M43, JAG, JAX(2R20), HELP19, JAX3

Table 2. Neat Ingredients

Energetic	Binder	Plasticizer	Stabilizer	Other
NC	NC	ATEC	AKARDIT II	$KB_{11}H_{14}$
NG	CAB	Paraplex G-59	EC	TNB
RDX	PDNPA	DEP	MC	DNP
RDX (Chinese)	hycar 4051	DEGDN		DMNP
CL 20	TPE	NG		triazole
HMX	CAB 500-5	triacetin		CNNQ
TNT	hycar 4054			CP
TNAZ	BEMO-BMMO/THF			
NQ	BAMO-BAMO/AMMO-BAMO			

Table 3. Propellant Composition

Propellant	Composition (approximate percentages)
JA2	59.9% NC, 14.9% NG, 24.8% DEGDN, 0.1% AKARDIT II
RPD351	49.5% NC, 40.9% NG, 7.2% DEGDN, 0.8% AKARDIT II, 1.5% KN
JAG	51.2% NC, 18% NG, 11.5% DEGDN, 0.7% AKARDIT II, 3.5% paraplex G-59, 15% RDX
JAX (2R20)	50.6% NC, 11.4% NG, 19.2% DEGDN, 0.7% AKARDIT II, 17.8% RDX
M44	52.9% NC, 43% NG, 2% EC, 2% DEP
M30	28% NC, 22.5% NG, 47.7% NQ, 1.5% EC
M9	57.6% NC, 39.9% NG, 0.75% EC, 1.5% KN, 0.35% ALC
M43	76% RDX, 12% CAB, 7.6% plasticizer, 4% NC, 0.4% EC
XM39	76% RDX, 12% CAB, 7.6% ATEC, 4% NC, 0.4% EC
M10	96% NC, 1% DPA, 1% KS, 1.5% water
HELP19	76% RDX, 11% CAB, 4% DANPE, 4% NC, 0.4% EC

5. CONCLUSIONS

PA-FTIR spectroscopy is an excellent technique for measuring spectra of propellant formulations and neat ingredients of these formulations. The spectra presented in this library are the initial spectra that have been measured for a larger library. These spectra have also been used to train a neural network that will be used to identify unknown formulations and quantify component concentrations for both quality control and aging studies. The result of preliminary training of the neural network were presented at the 1995 Federation of Analytical Chemists and Spectroscopist Societies meeting. This spectral library should be useful for identification of unknowns and also useful as a guide for determination of changes in propellant composition during storage.

A planned conversion of our spectrometer to step-scan operation will allow for spectral depth profiling of propellant surfaces. This will permit measurement of ingredient concentration profiles near the surface of propellant grains, providing valuable information that may be characteristic of manufacture and/or storage condition.

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6. REFERENCES

Dittmar, R. M., R. A. Palmer, and R. O. Carter III. "Fourier Transform Photoacoustic Spectroscopy of Polymer." Applied Spectroscopy Reviews, vol. 29, p. 171, 1994.

McClelland, J. F., R. W. Jones, S. Luo, and L. M. Seaverson. "A Practical Guide to FT-IR Photoacoustic Spectroscopy." Practical Sampling Technique for Infrared Analysis, edited by Patricia B. Coleman, ch. 5, 1993.

Palmer, Richard A. "Photoacoustic and Photothermal Spectroscopies." Determination of Electronic and Optical Properties, edited by Bryant W. Rossiter and Roger C. Baetzold, Physical Methods of Chemistry Series, Second edition, vol. 8, ch. 2, 1993.

Palmer, Richard A. "Step-Scan FT-IR: A Versatile Tool for Time- and Phase-Resolved Vibrational Spectroscopy." Spectroscopy, vol. 8, no. 2, p. 26, 1993.

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APPENDIX A:

SPECTRA OF FORMULATIONS

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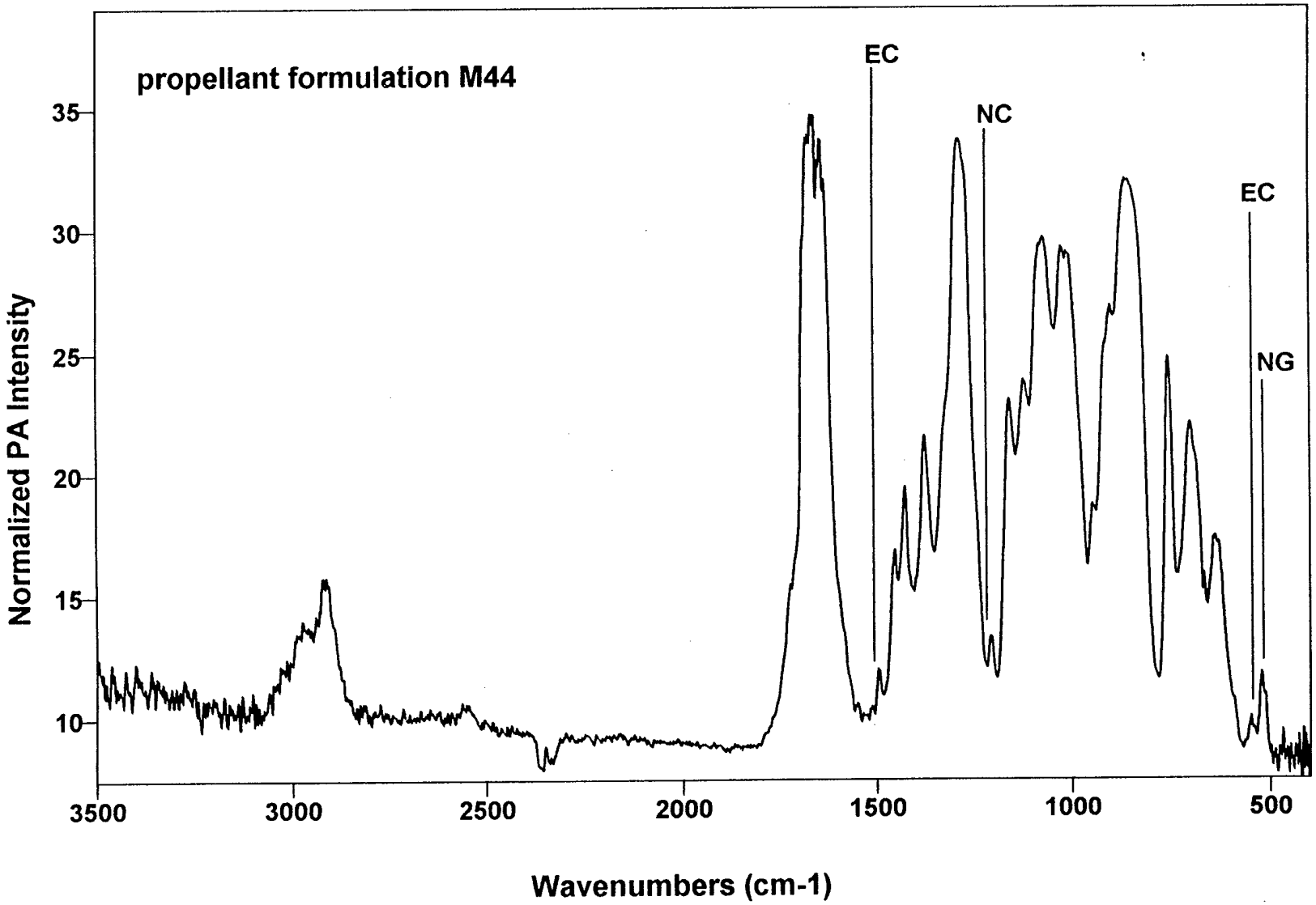
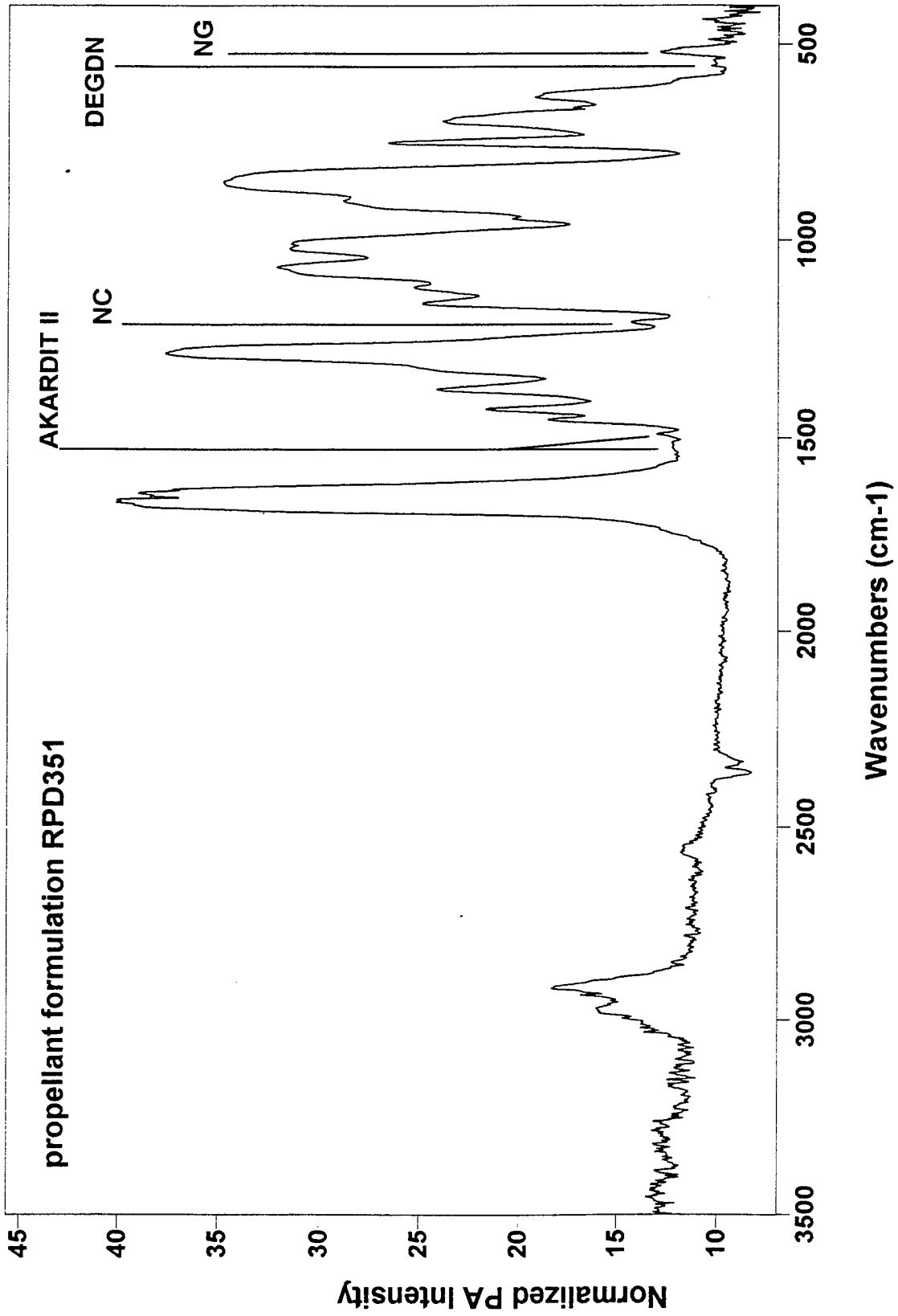

propellant formulation M44
EC
NC
EC
NG
Normalized PA Intensity
35
30
25
20
15
10
3500
3000
2500
2000
1500
1000
500
Wavenumbers (cm-1)


propellant formulation RPD351
AKARDIT II
NC
DEGDN
NG
Normalized PA Intensity
Wavenumbers (cm-1)
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40
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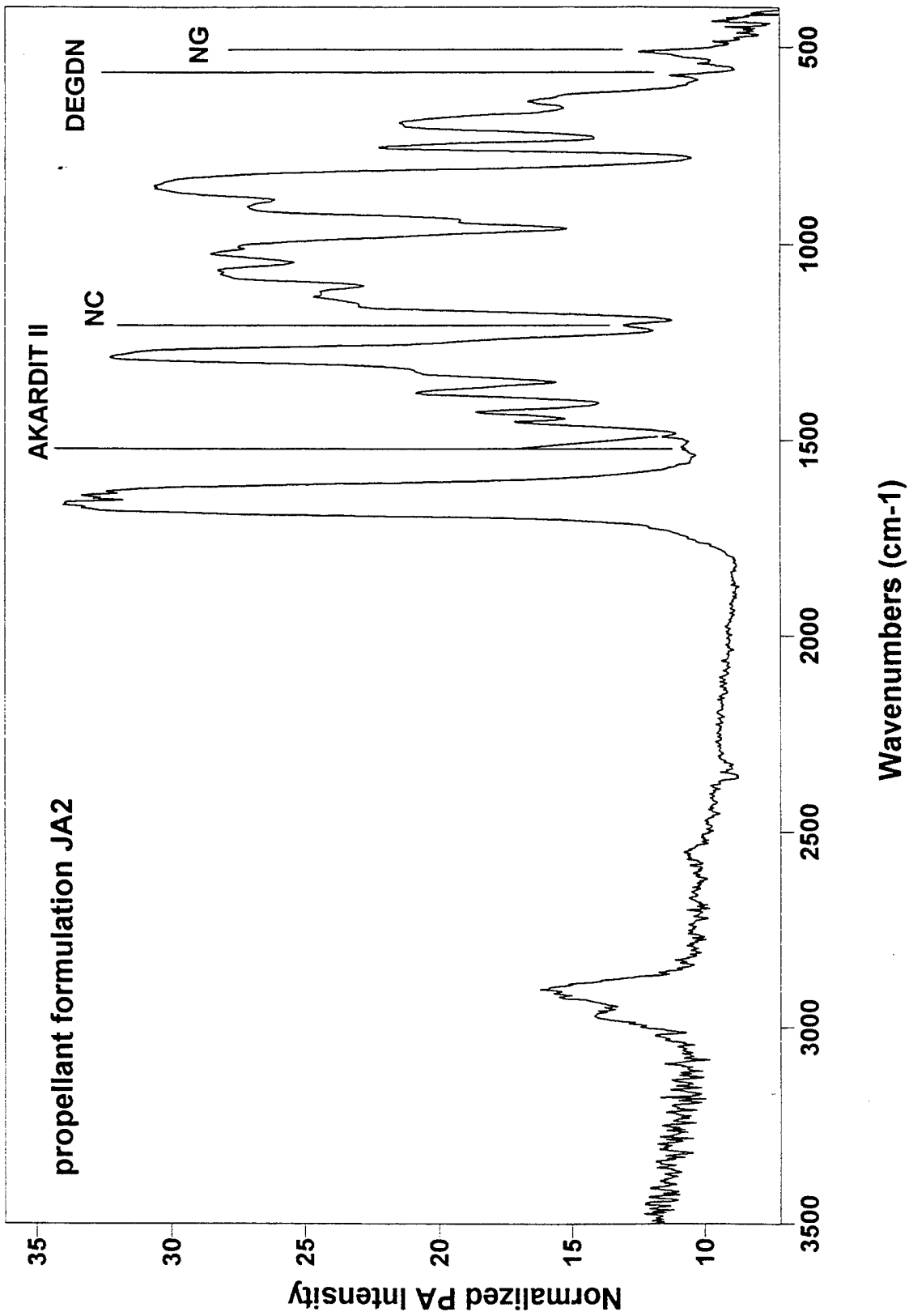
30
25
20
15
10
3500
3000
2500
2000
1500
1000
500


propellant formulation JA2
AKARDIT II
NC
DEGDN
NG
Normalized PA Intensity
Wavenumbers (cm-1)
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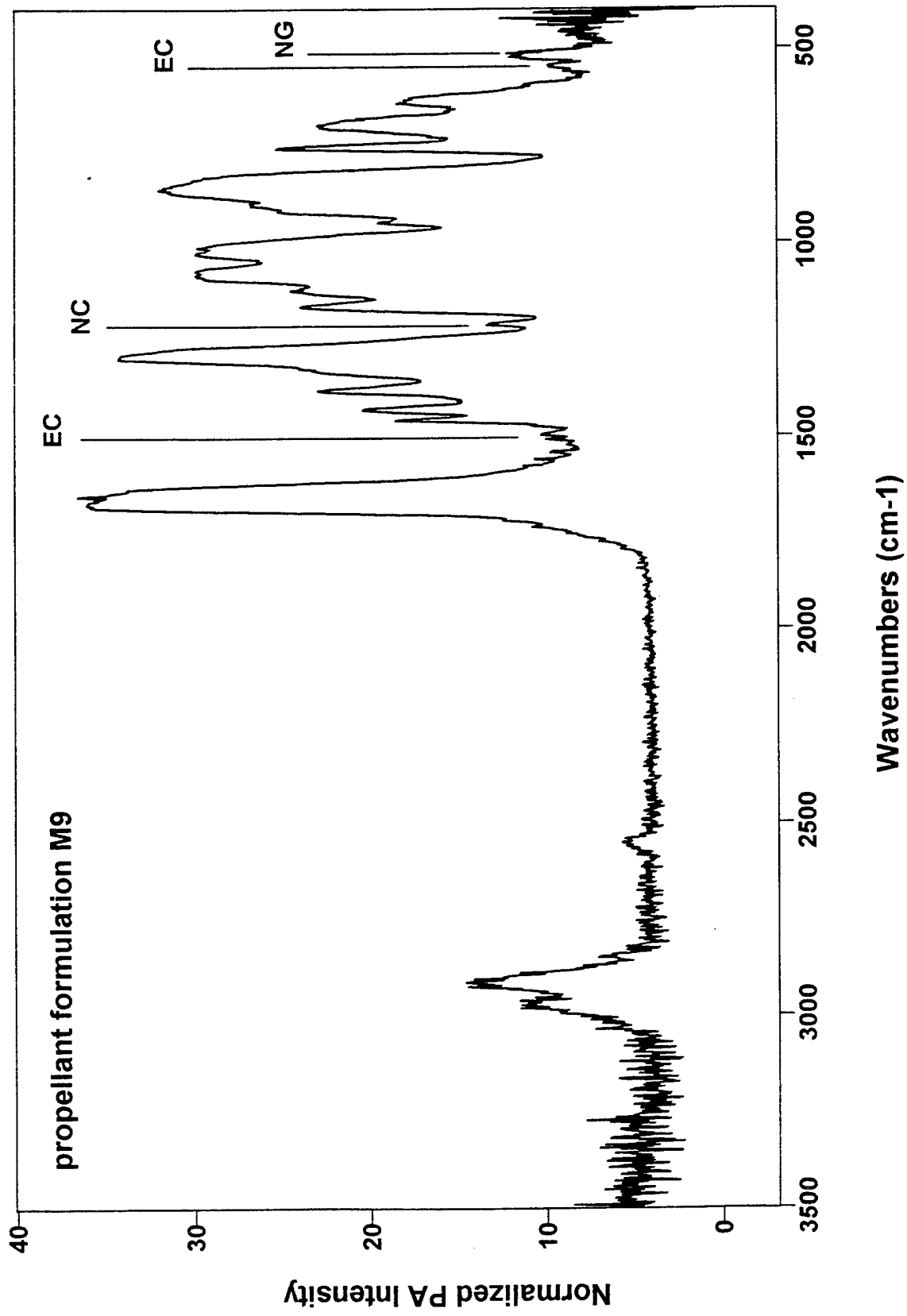
30
25
20
15
10
3500
3000
2500
2000
1500
1000
500


propellant formulation M9
EC
NC
EC
NG
Normalized PA Intensity
Wavenumbers (cm-1)
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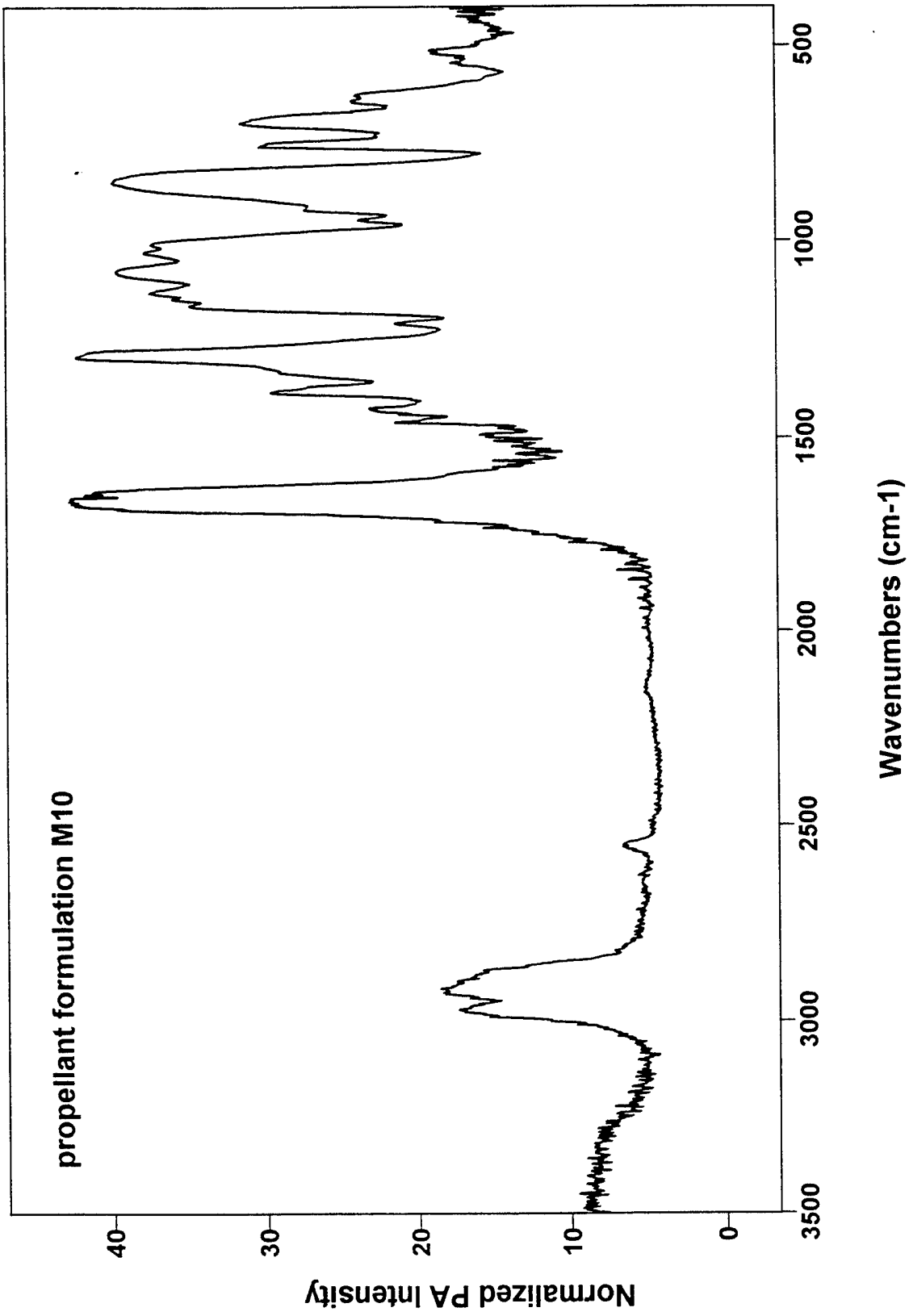
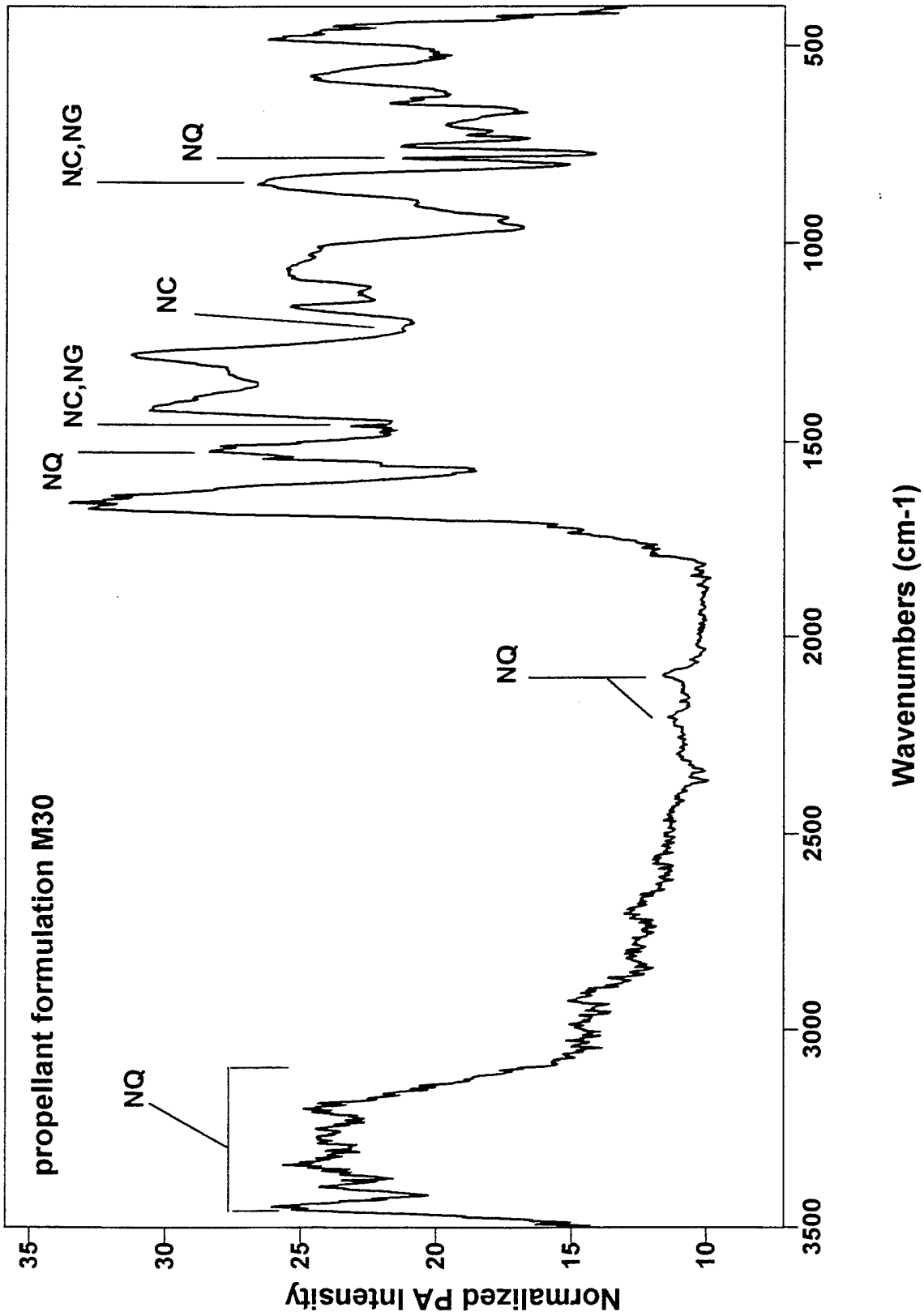
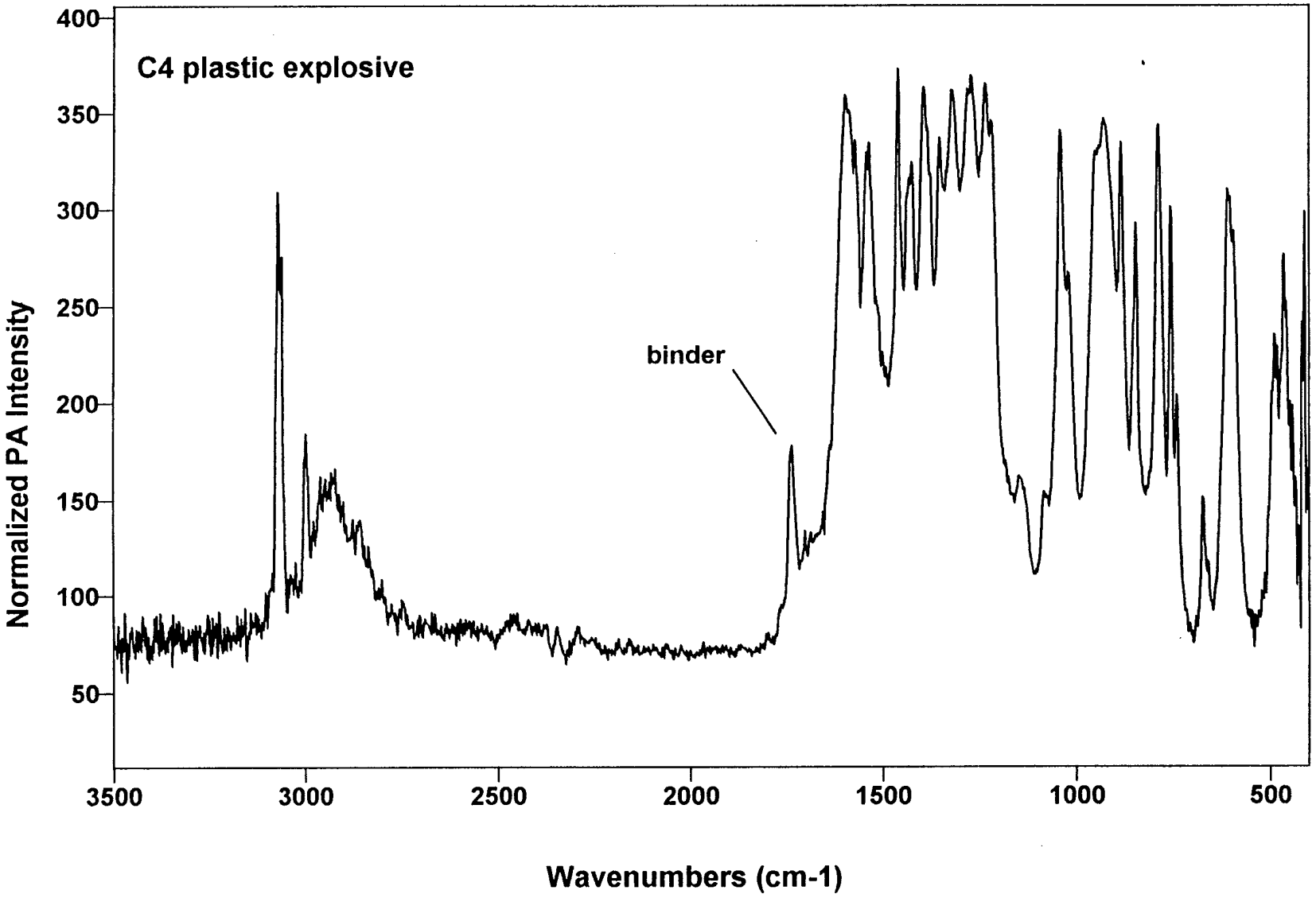
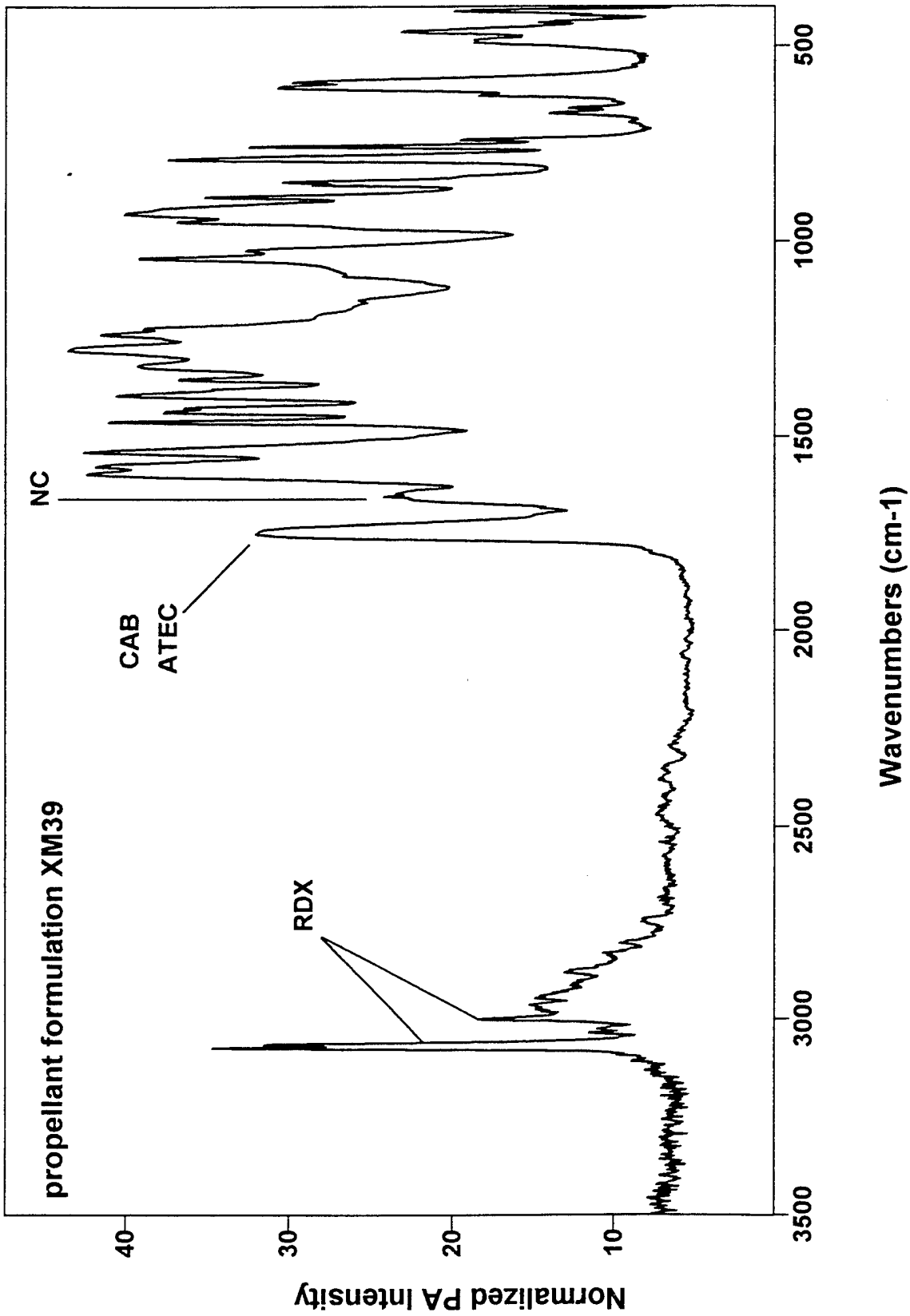
30
20
10
0
3500
3000
2500
2000
1500
1000
500


propellant formulation M10
Normalized PA Intensity
0
10
20
30
40
3500
3000
2500
2000
1500
1000
500
Wavenumbers (cm-1)


propellant formulation M30
Normalized PA Intensity
Wavenumbers (cm-1)
NQ
NQ
NC,NG
NC
NC,NG
NQ
NQ
35
30
25
20
15
10
3500
3000
2500
2000
1500
1000
500


C4 plastic explosive
binder
Normalized PA Intensity
400
350
300
250
200
150
100
50
3500
3000
2500
2000
1500
1000
500
Wavenumbers (cm-1)


propellant formulation XM39
NC
CAB
ATEC
RDX
Normalized PA Intensity
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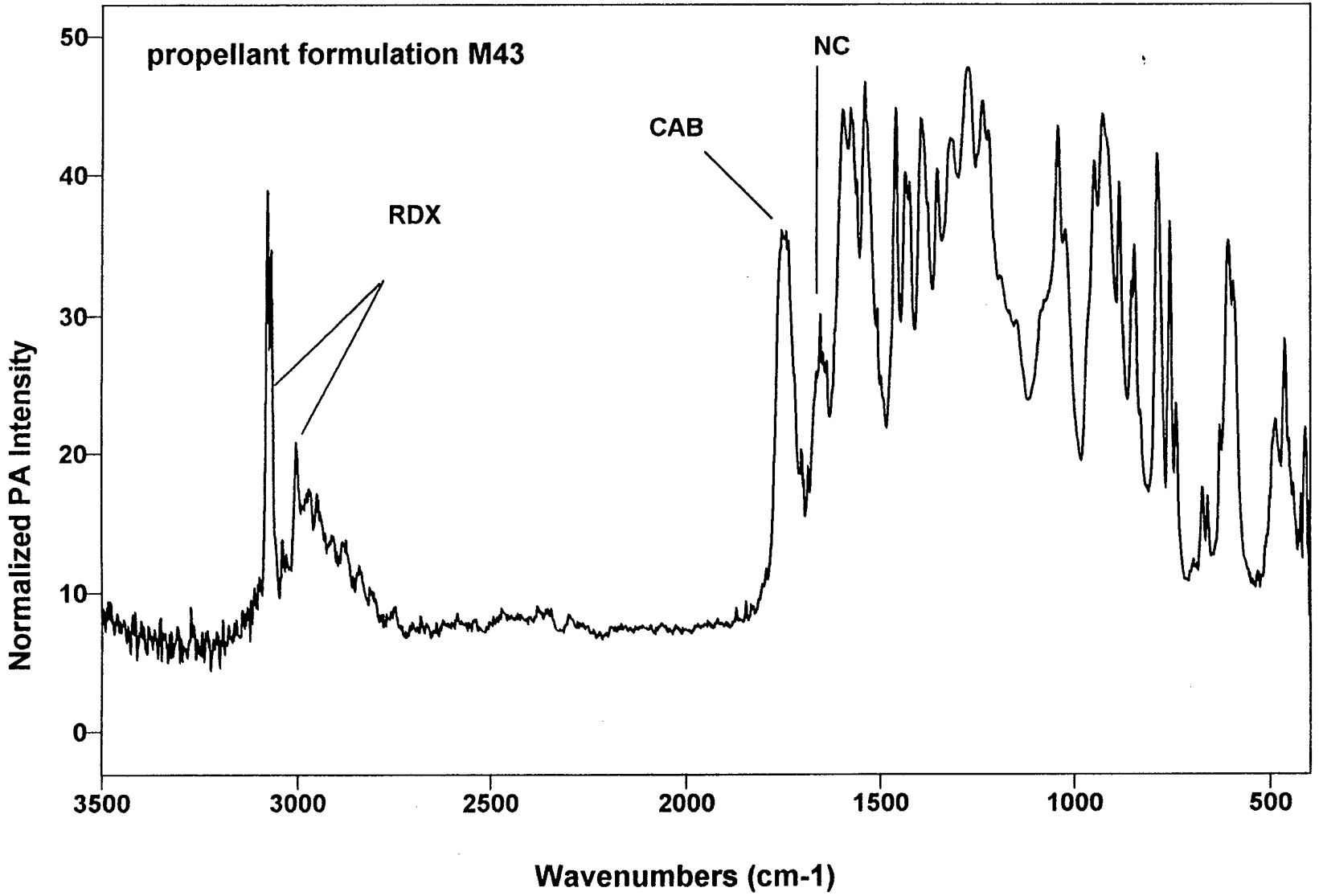
30
20
10
3500
3000
2500
2000
1500
1000
500
Wavenumbers (cm-1)


propellant formulation M43
NC
CAB
RDX
Normalized PA Intensity
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20
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3000
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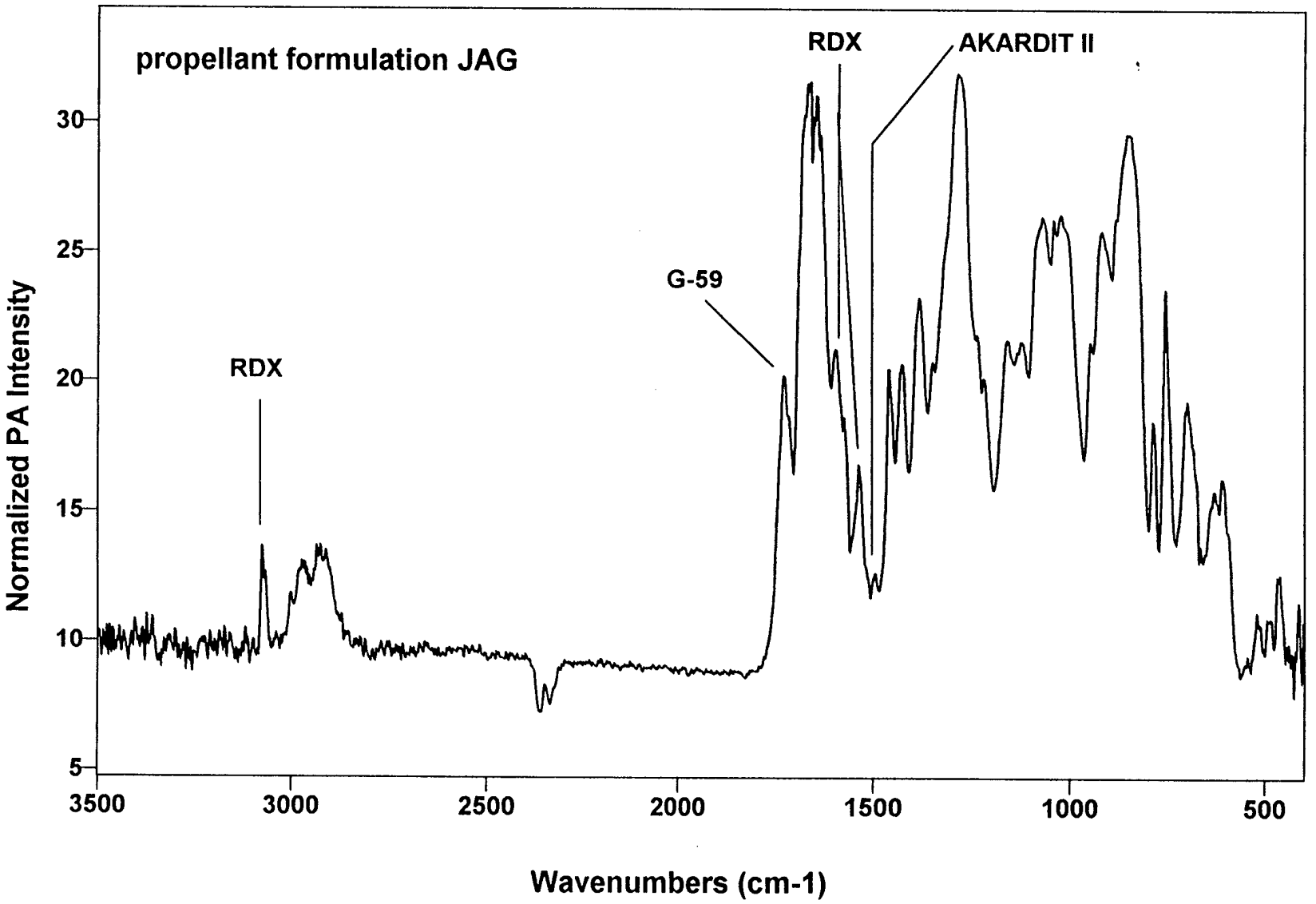
2000
1500
1000
500
Wavenumbers (cm-1)


propellant formulation JAG
RDX
AKARDIT II
G-59
RDX
Normalized PA Intensity
30
25
20
15
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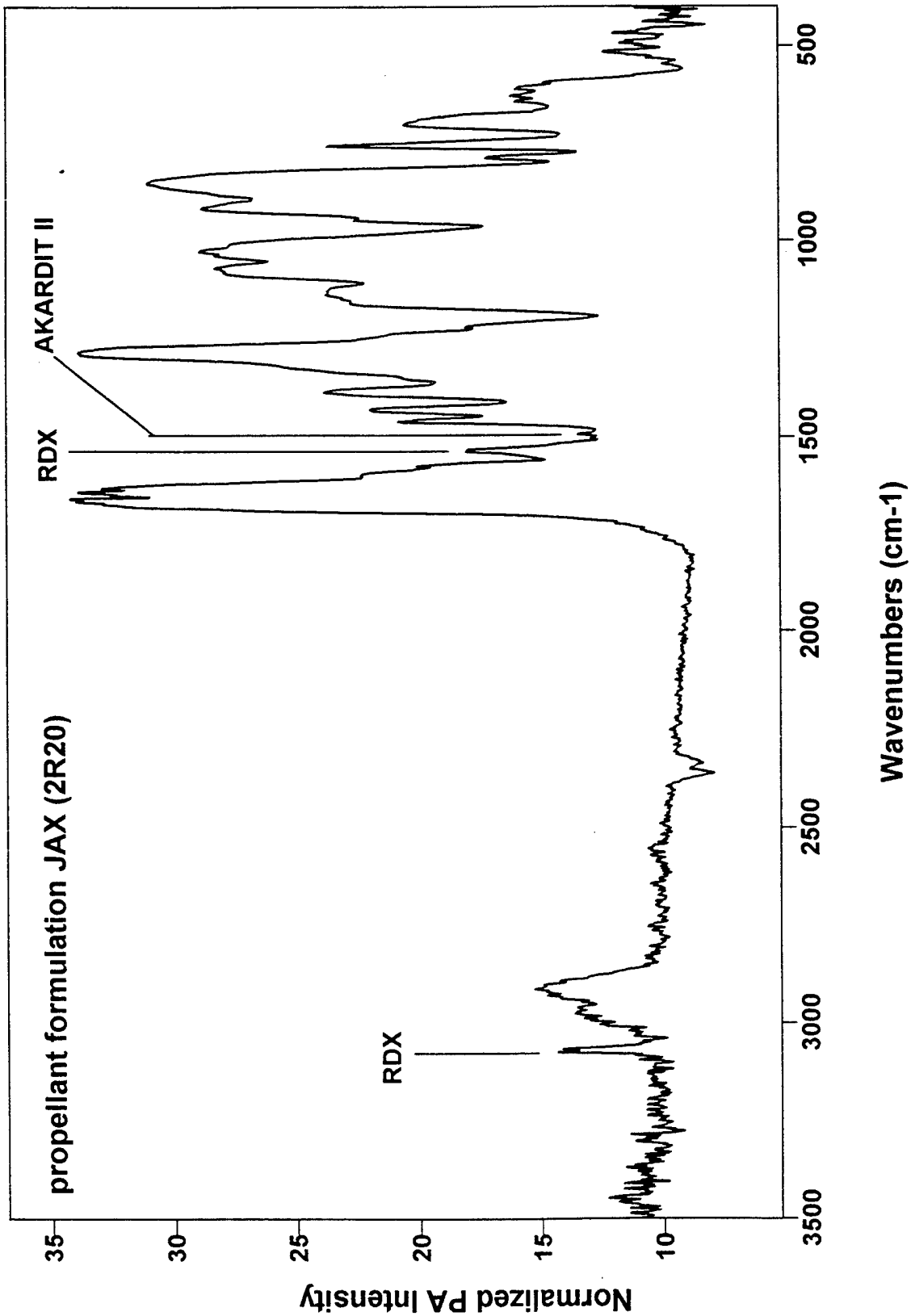
5
3500
3000
2500
2000
1500
1000
500
Wavenumbers (cm-1)


propellant formulation JAX (2R20)
RDX
AKARDIT II
RDX
Normalized PA Intensity
Wavenumbers (cm-1)
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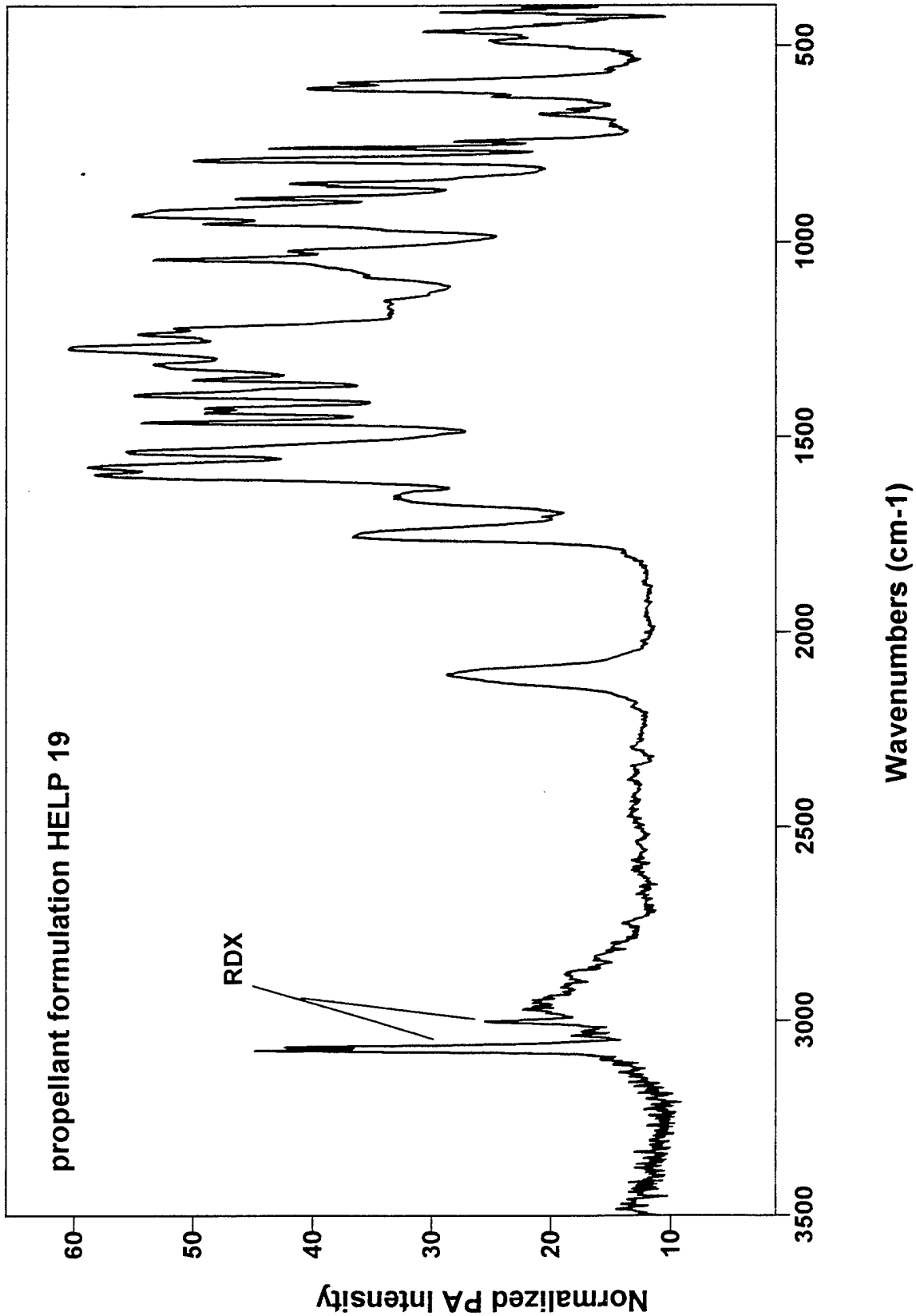
25
20
15
10
3500
3000
2500
2000
1500
1000
500


propellant formulation HELP 19
RDX
Normalized PA Intensity
Wavenumbers (cm-1)
60
50
40
30
20
10
3500
3000
2500
2000
1500
1000
500

APPENDIX B:

SPECTRA OF NEAT ENERGETIC MATERIALS

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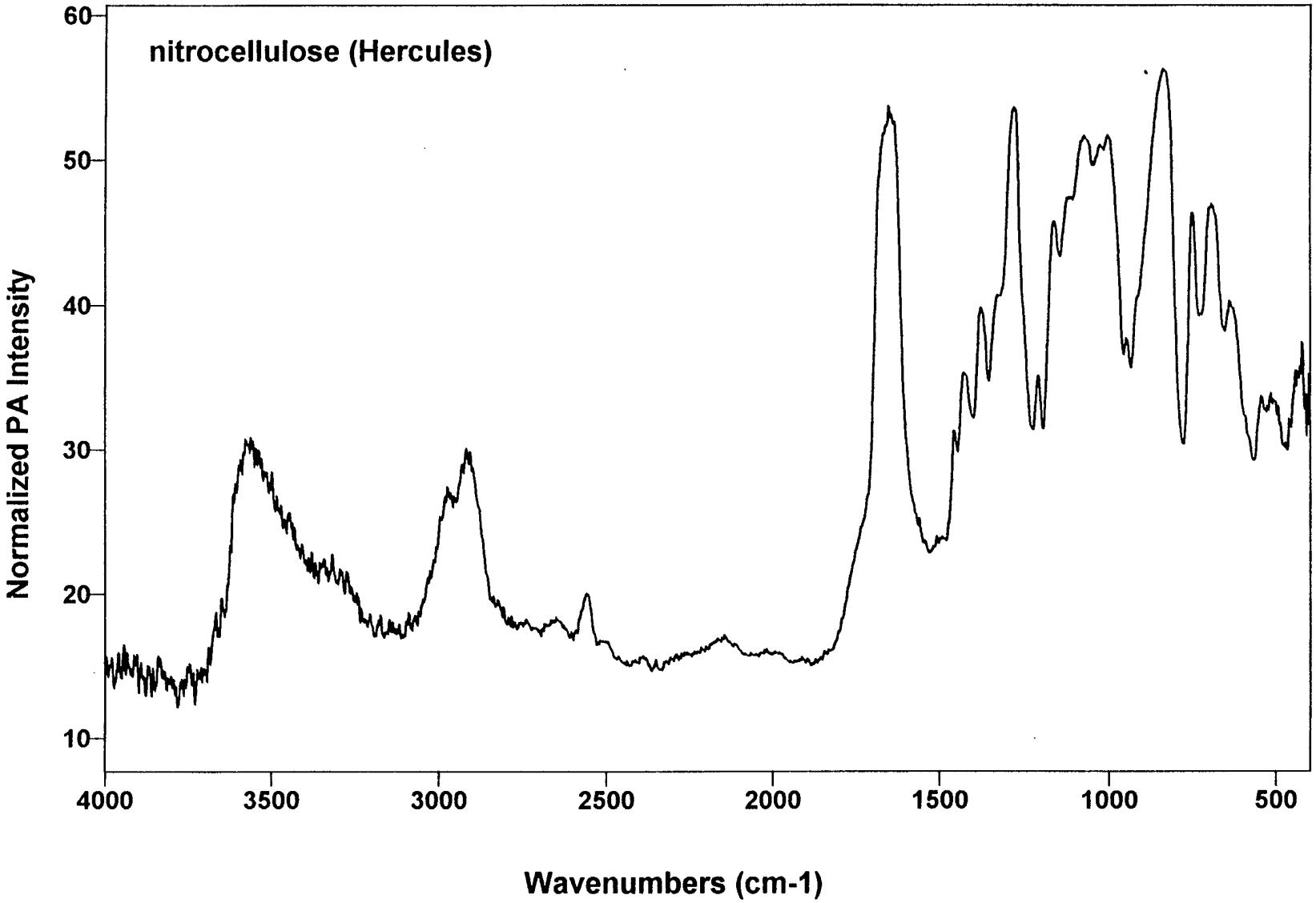
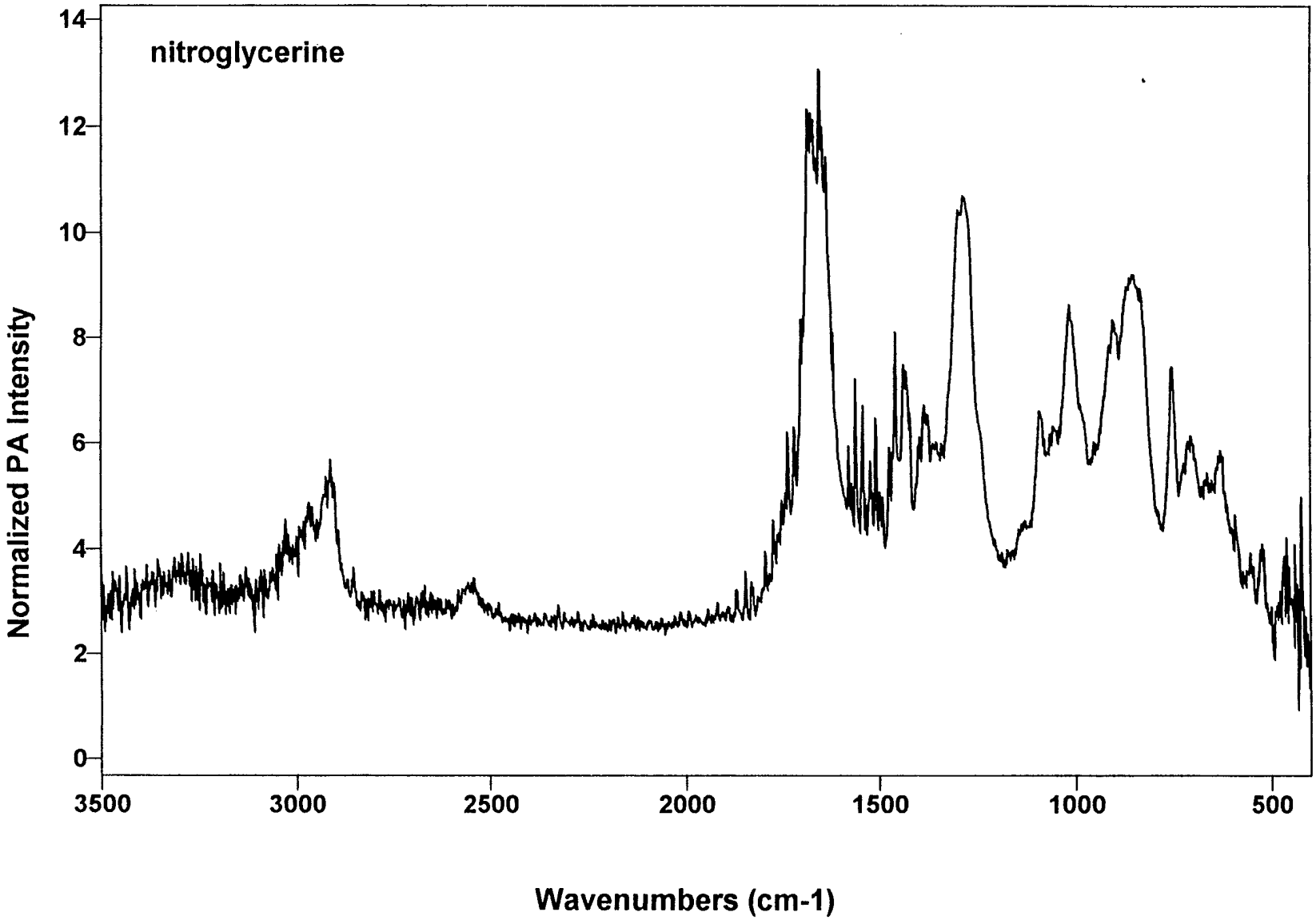
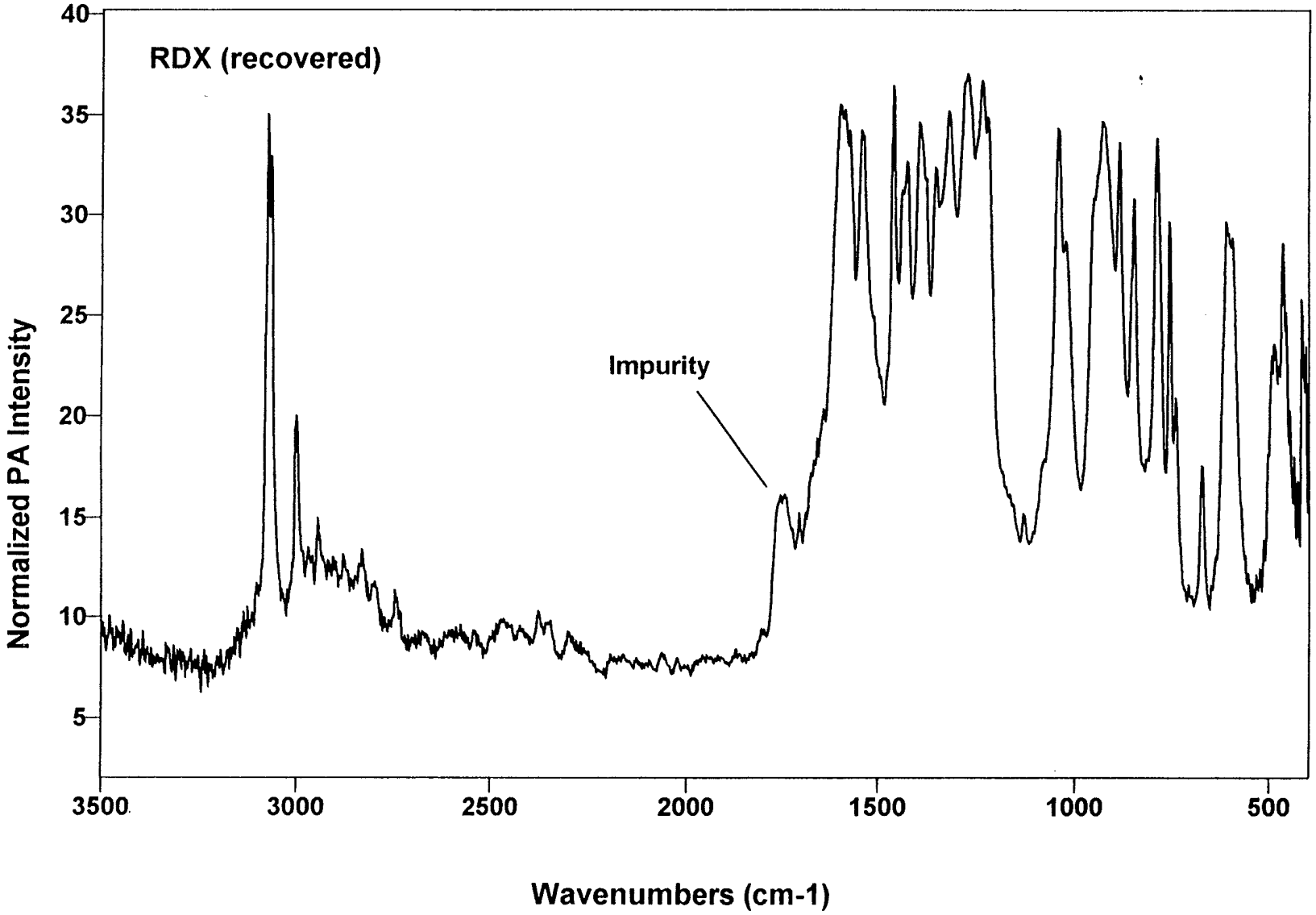

nitrocellulose (Hercules)
Normalized PA Intensity
60
50
40
30
20
10
4000
3500
3000
2500
2000
1500
1000
500
Wavenumbers (cm-1)


nitroglycerine
Normalized PA Intensity
14
12
10
8
6
4
2
0
3500
3000
2500
2000
1500
1000
500
Wavenumbers (cm-1)


RDX (recovered)
Impurity
Normalized PA Intensity
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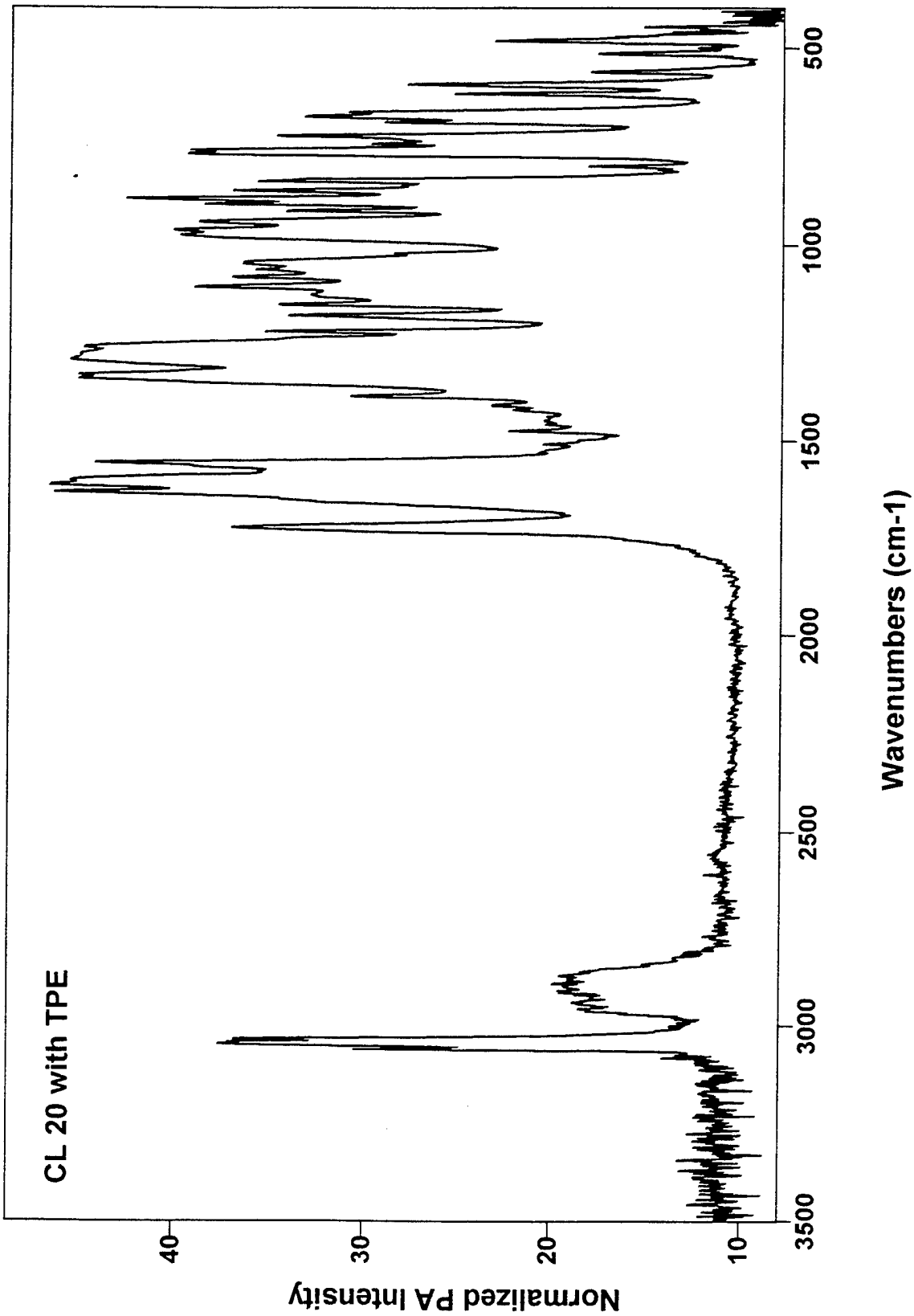
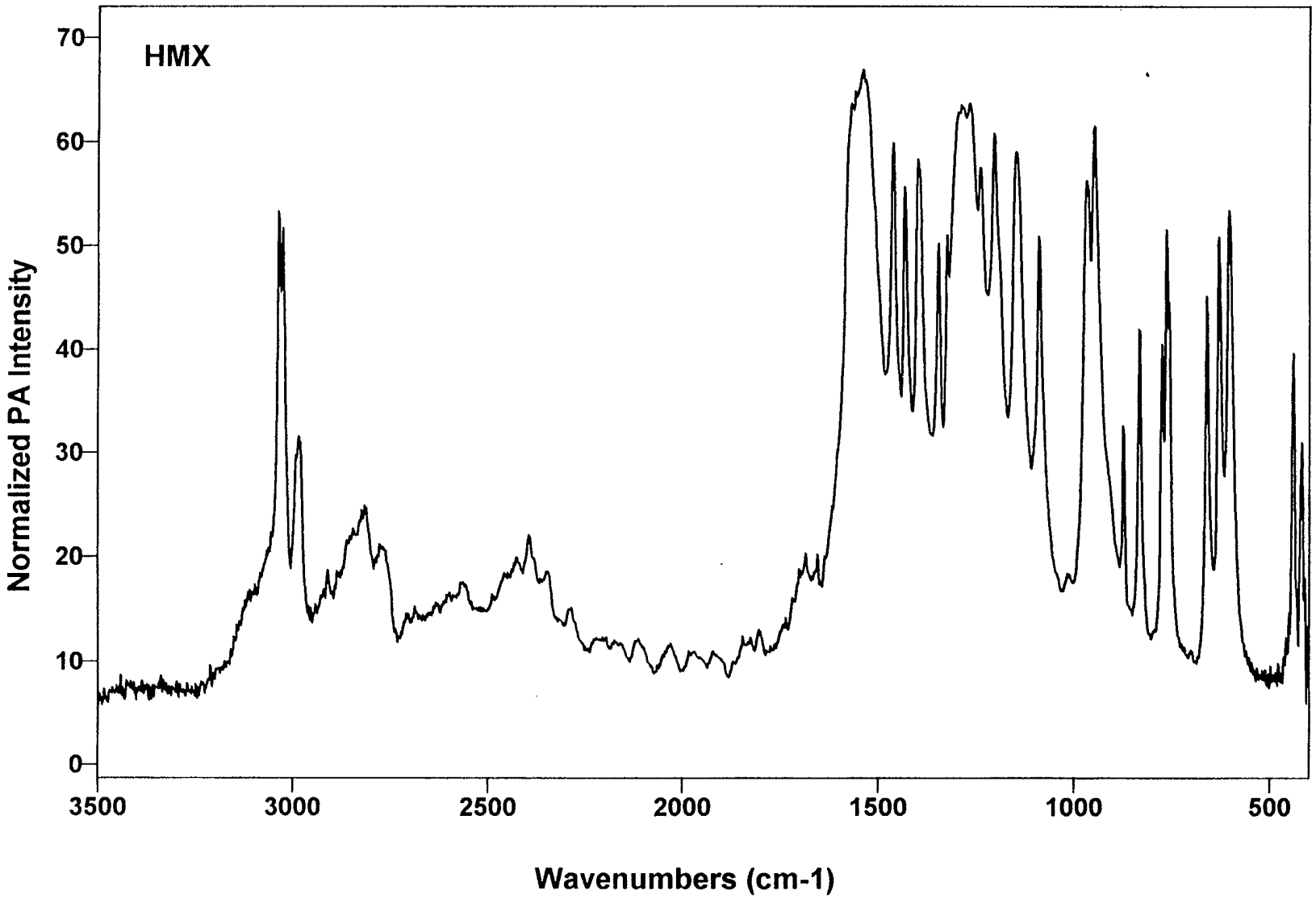
35
30
25
20
15
10
5
3500
3000
2500
2000
1500
1000
500
Wavenumbers (cm-1)


CL 20 with TPE
Normalized PA Intensity
40
30
20
10
3500
3000
2500
2000
1500
1000
500
Wavenumbers (cm-1)


HMX
Normalized PA Intensity
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60
50
40
30
20
10
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3500
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2500
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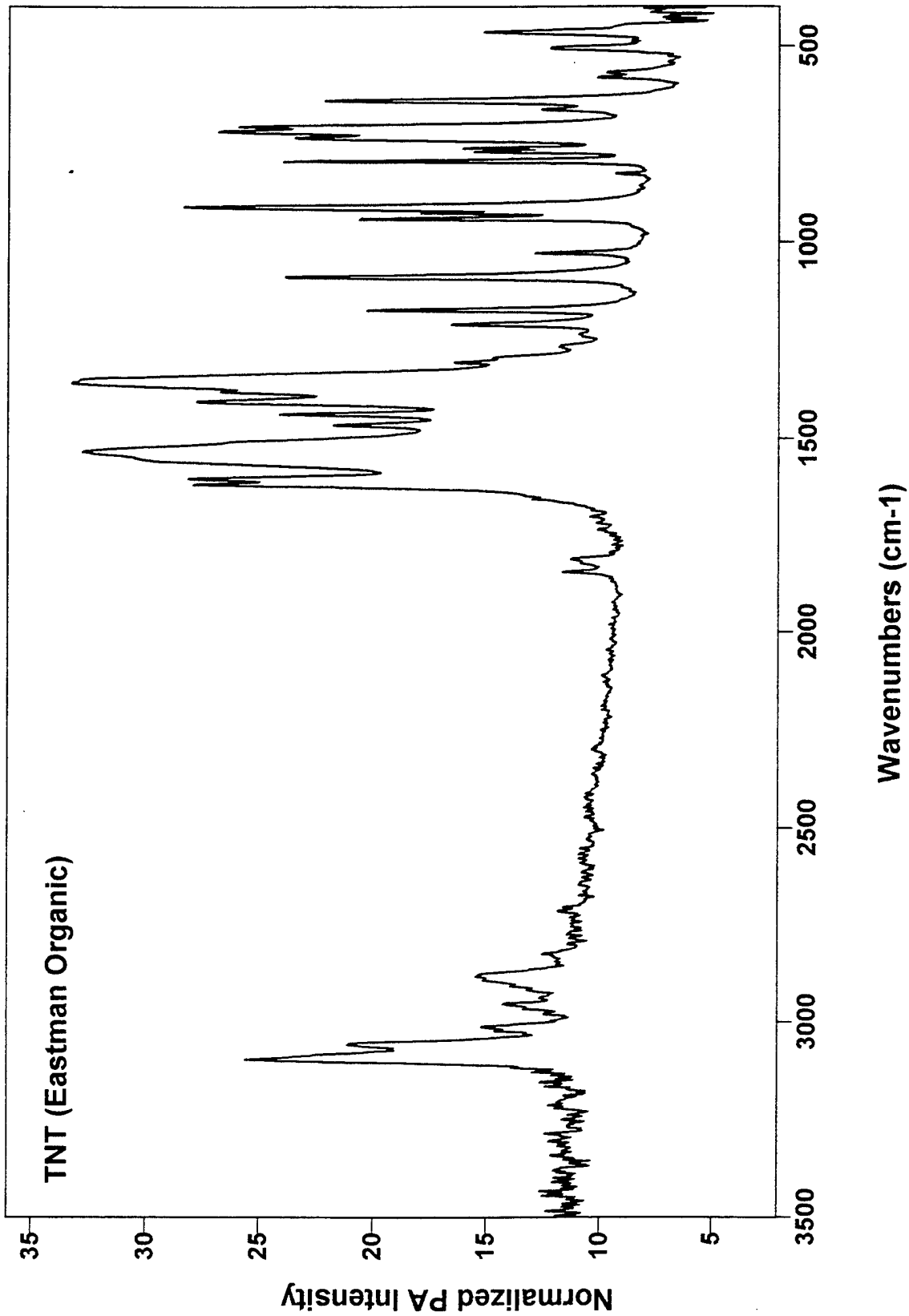
1000
500
Wavenumbers (cm-1)


TNT (Eastman Organic)
Normalized PA Intensity
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30
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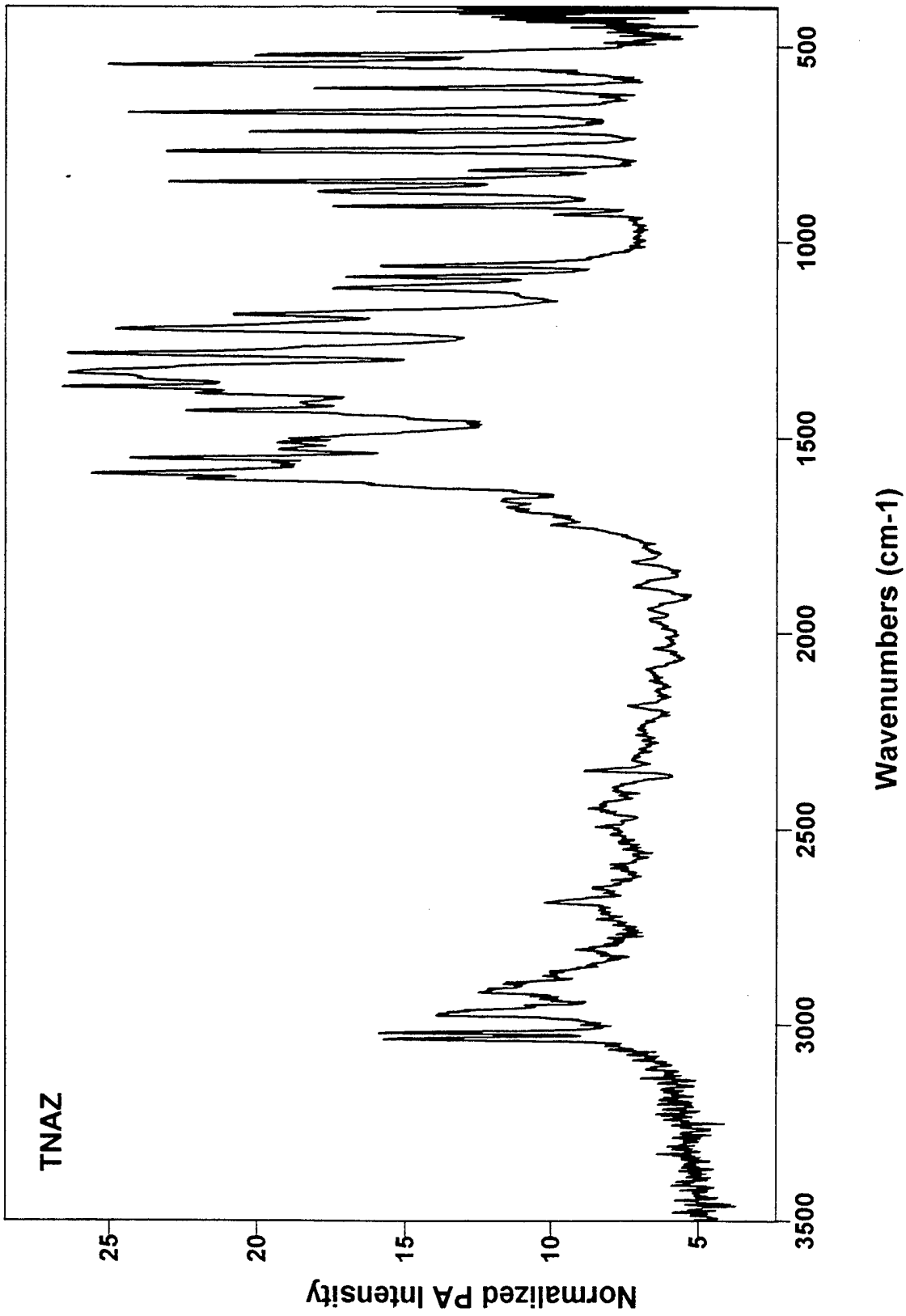
20
15
10
5
3500
3000
2500
2000
1500
1000
500
Wavenumbers (cm-1)


TNAZ
Normalized PA Intensity
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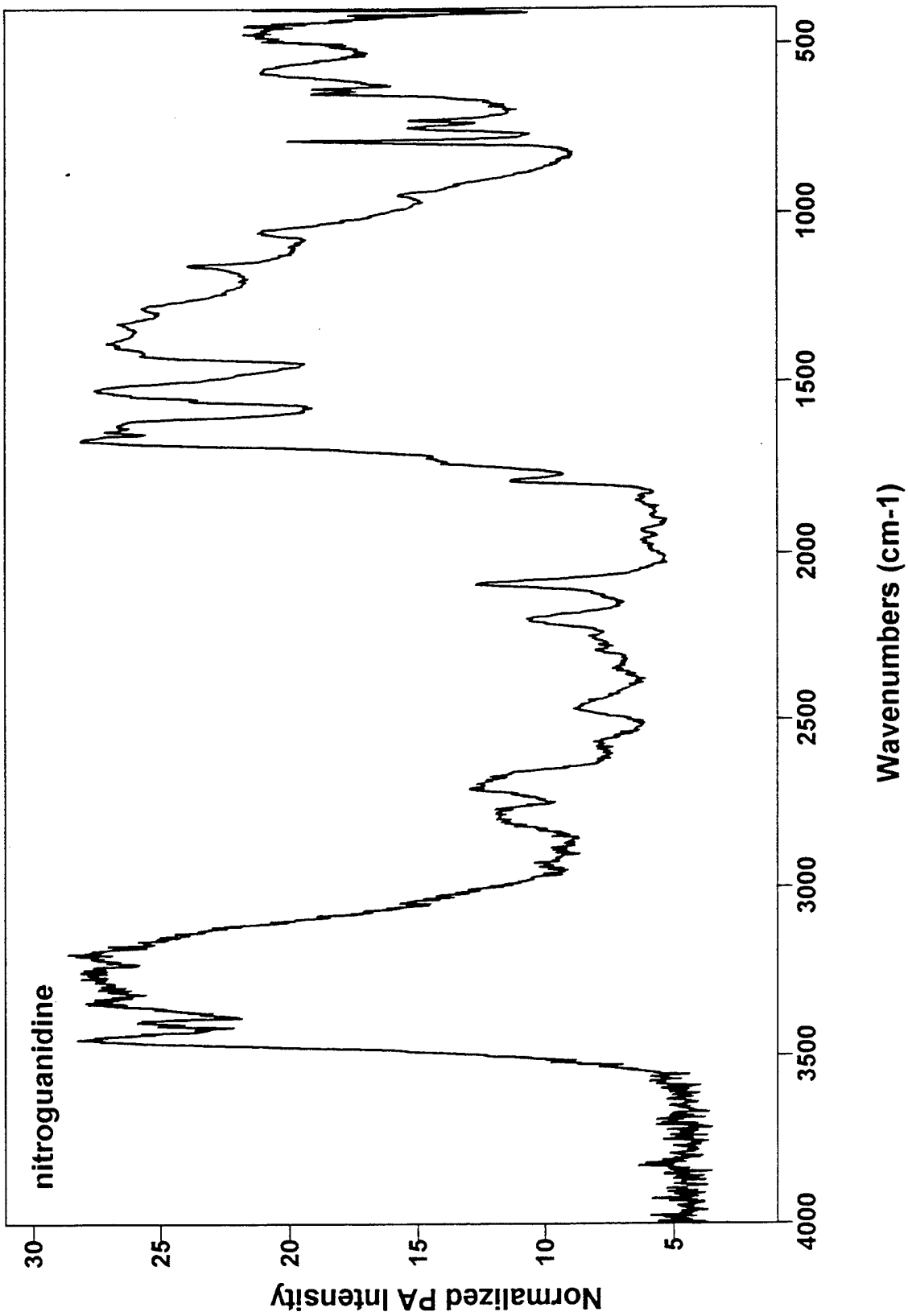
20
15
10
5
3500
3000
2500
2000
1500
1000
500
Wavenumbers (cm-1)


nitroguanidine
Normalized PA Intensity
30
25
20
15
10
5
4000
3500
3000
2500
2000
1500
1000
500
Wavenumbers (cm-1)

APPENDIX C:

SPECTRA OF BINDERS

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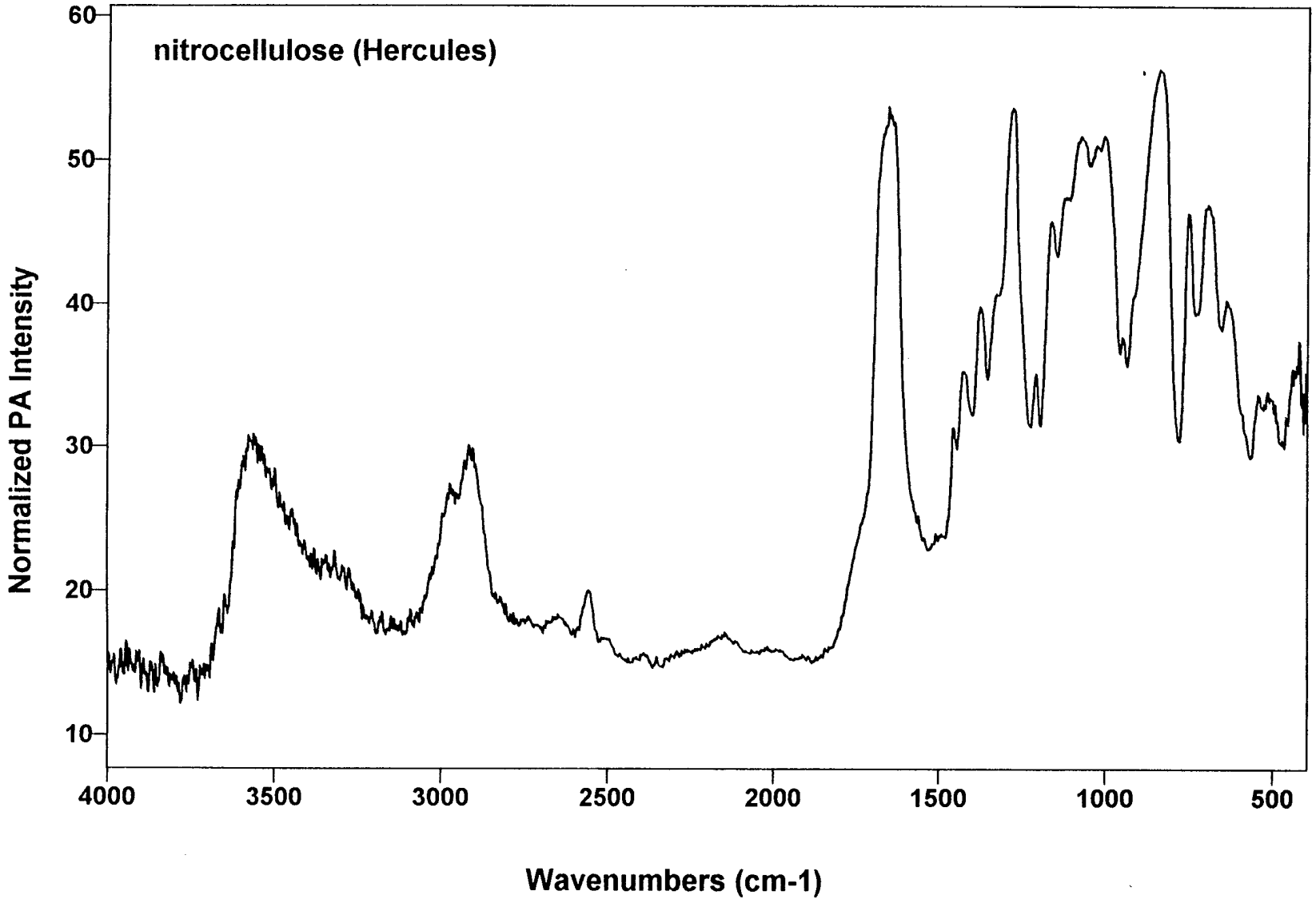
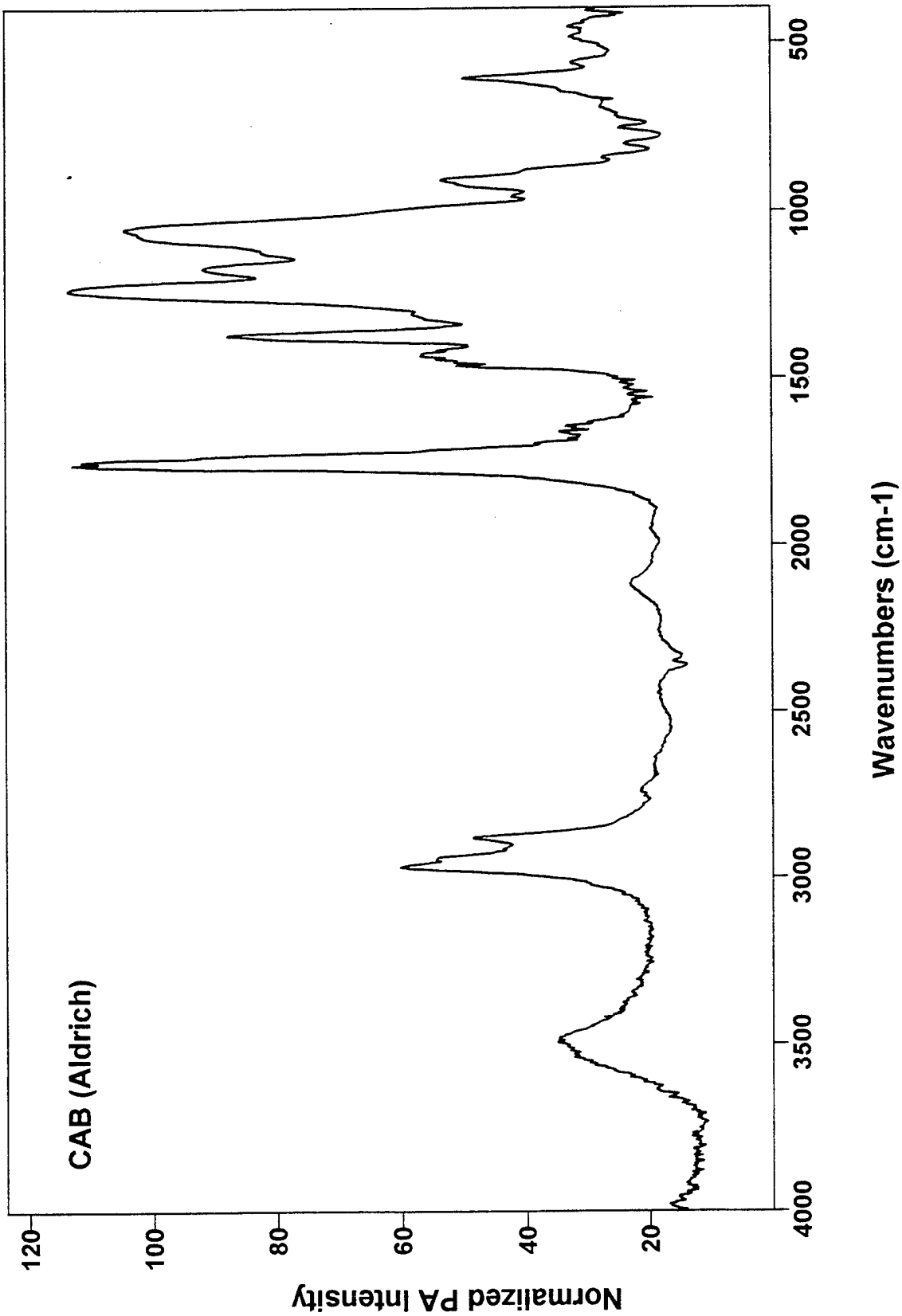
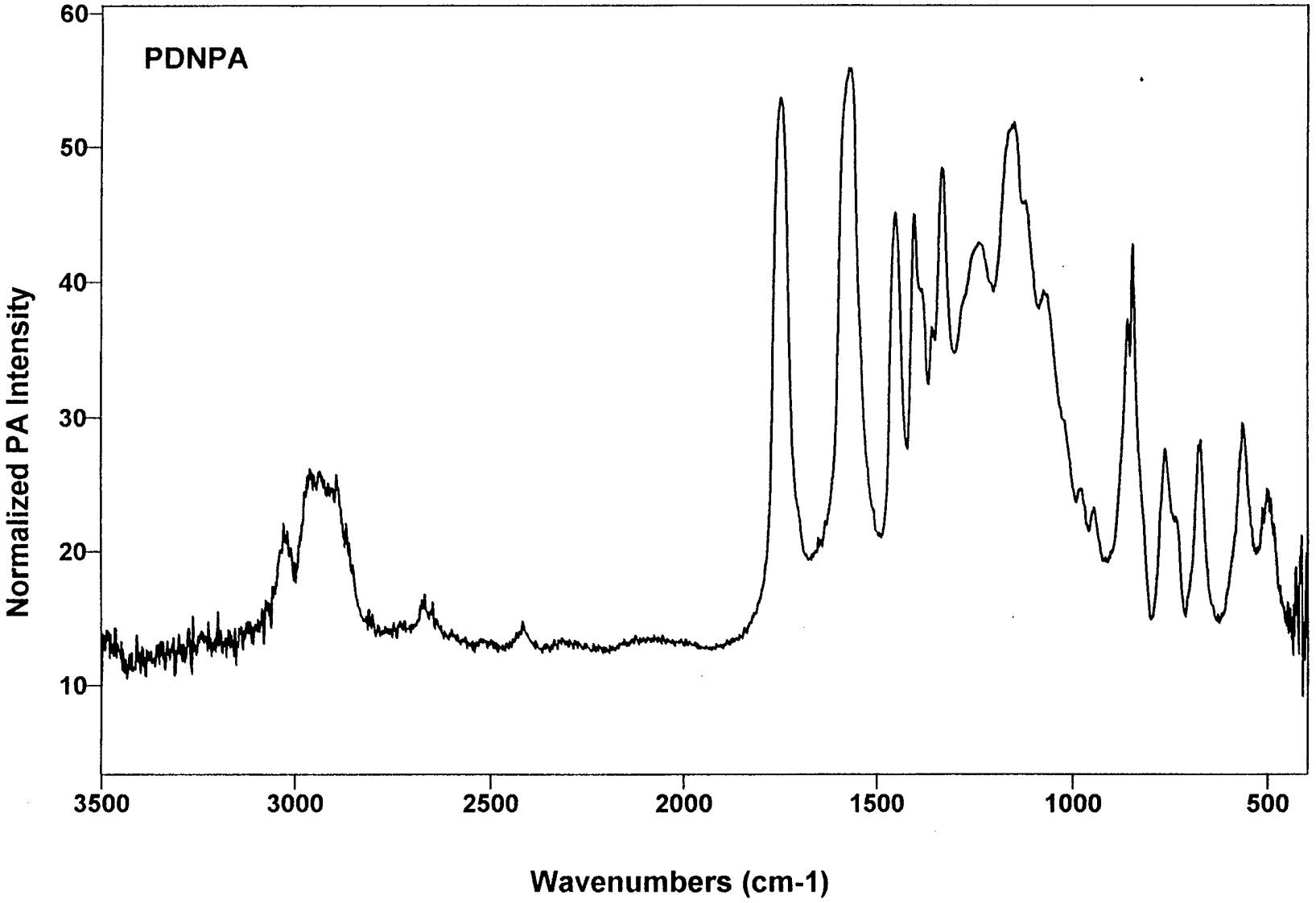

nitrocellulose (Hercules)
Normalized PA Intensity
60
50
40
30
20
10
4000
3500
3000
2500
2000
1500
1000
500
Wavenumbers (cm-1)


CAB (Aldrich)
Normalized PA Intensity
120
100
80
60
40
20
4000
3500
3000
2500
2000
1500
1000
500
Wavenumbers (cm-1)


PDNPA
Normalized PA Intensity
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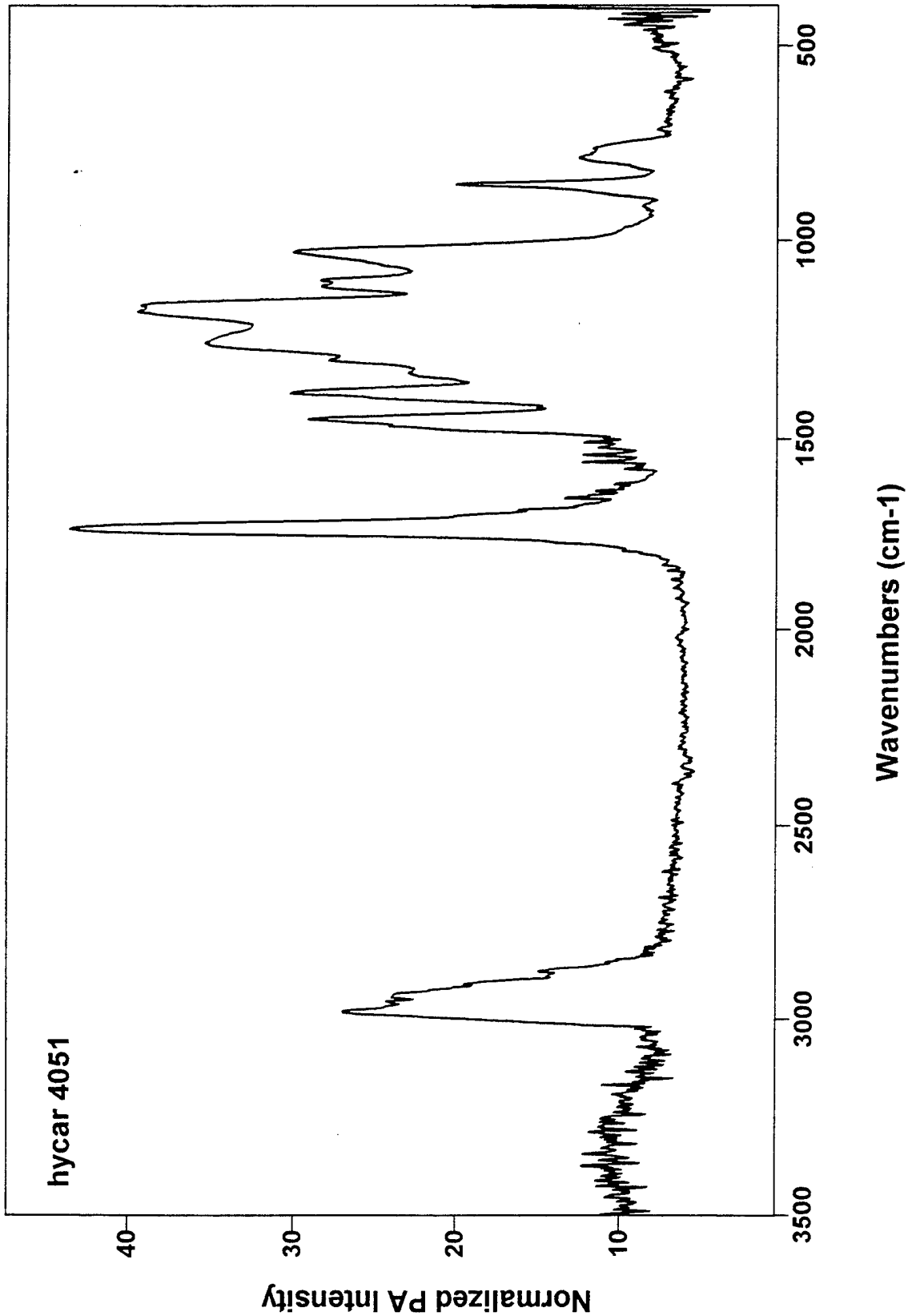
50
40
30
20
10
3500
3000
2500
2000
1500
1000
500
Wavenumbers (cm-1)


hycar 4051
Normalized PA Intensity
40
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2500
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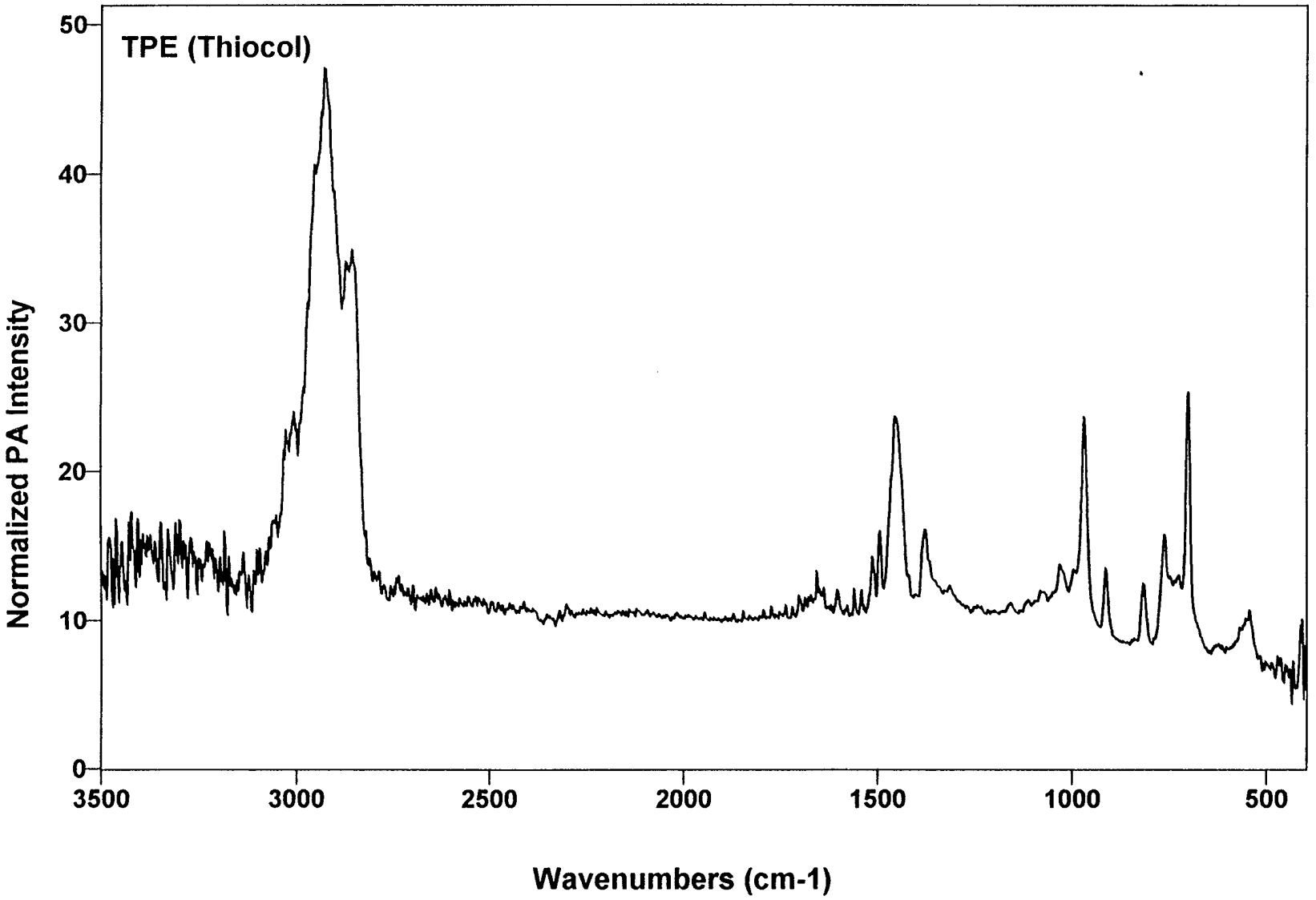
Wavenumbers (cm-1)


TPE (Thiocol)
Normalized PA Intensity
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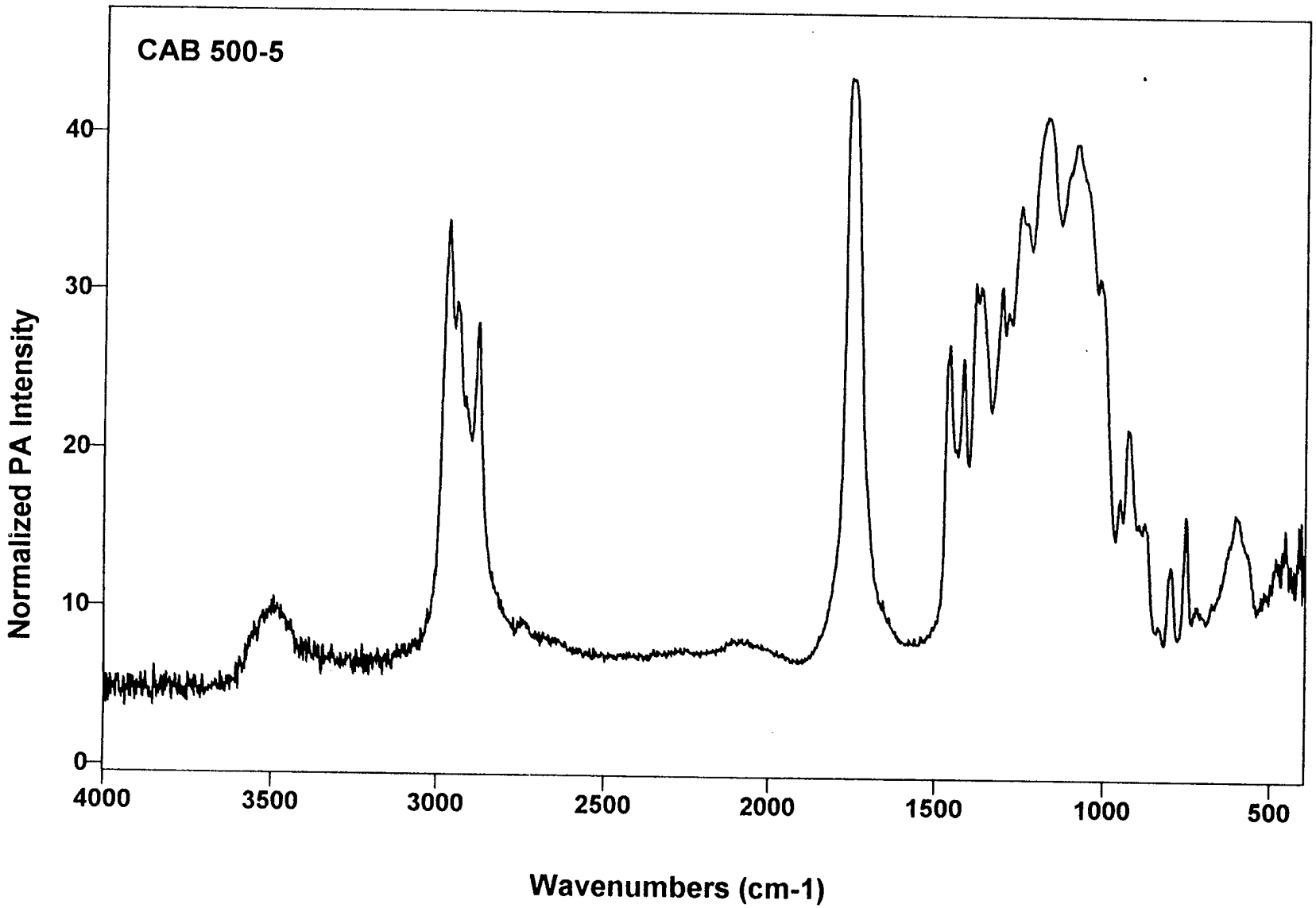
40
30
20
10
0
3500
3000
2500
2000
1500
1000
500
Wavenumbers (cm-1)


CAB 500-5
Normalized PA Intensity
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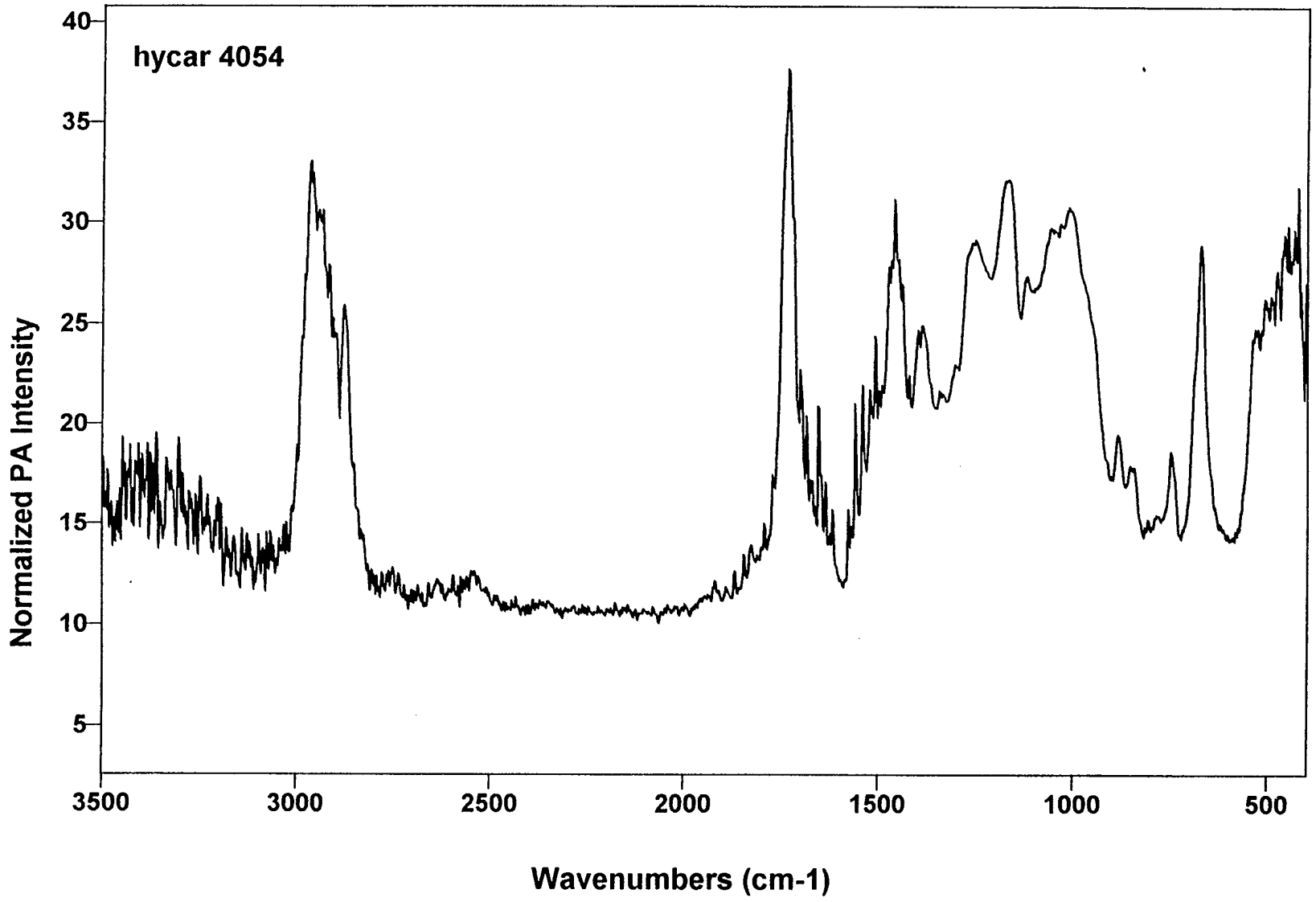
10
20
30
40
4000
3500
3000
2500
2000
1500
1000
500
Wavenumbers (cm-1)


hycar 4054
Normalized PA Intensity
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35
30
25
20
15
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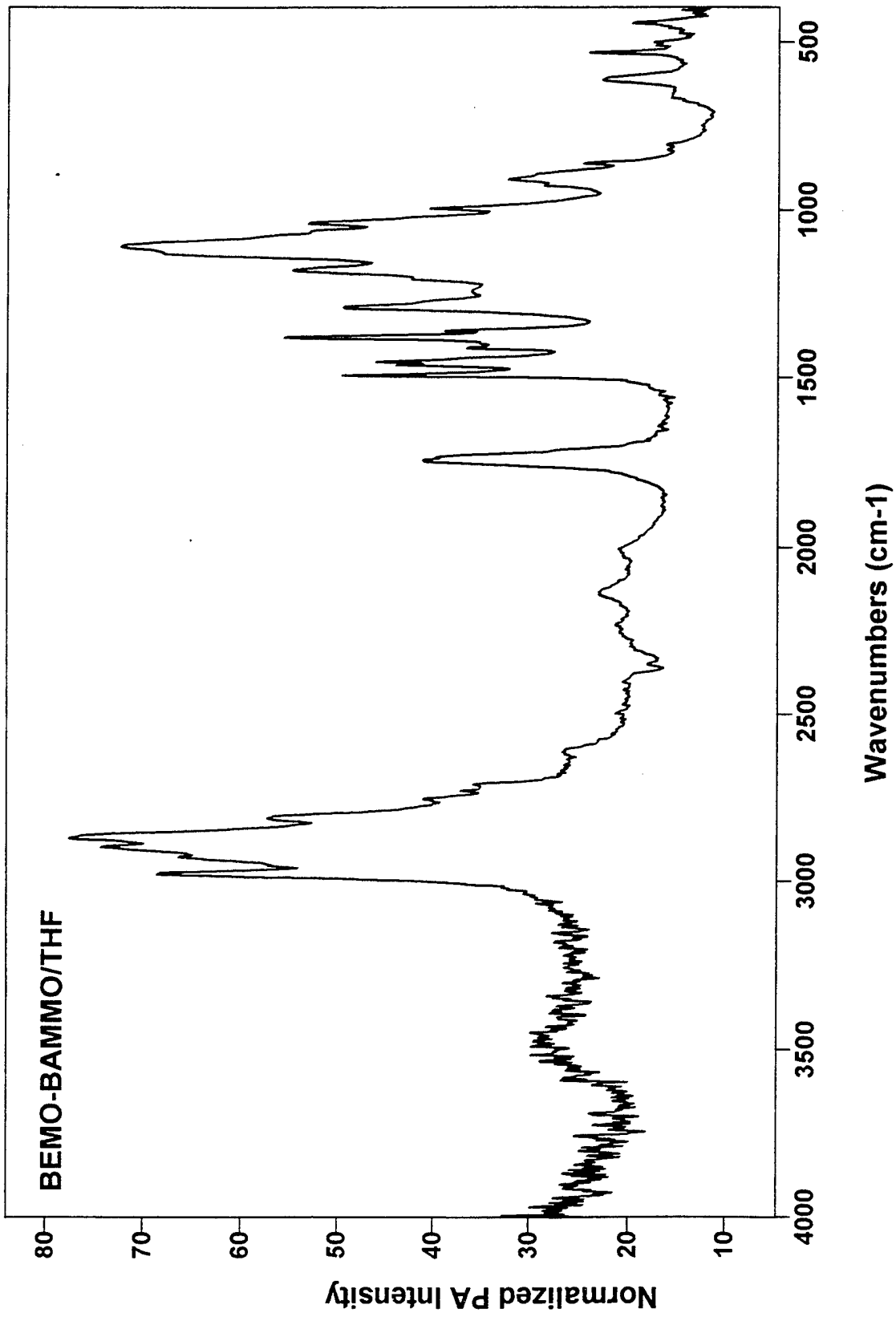
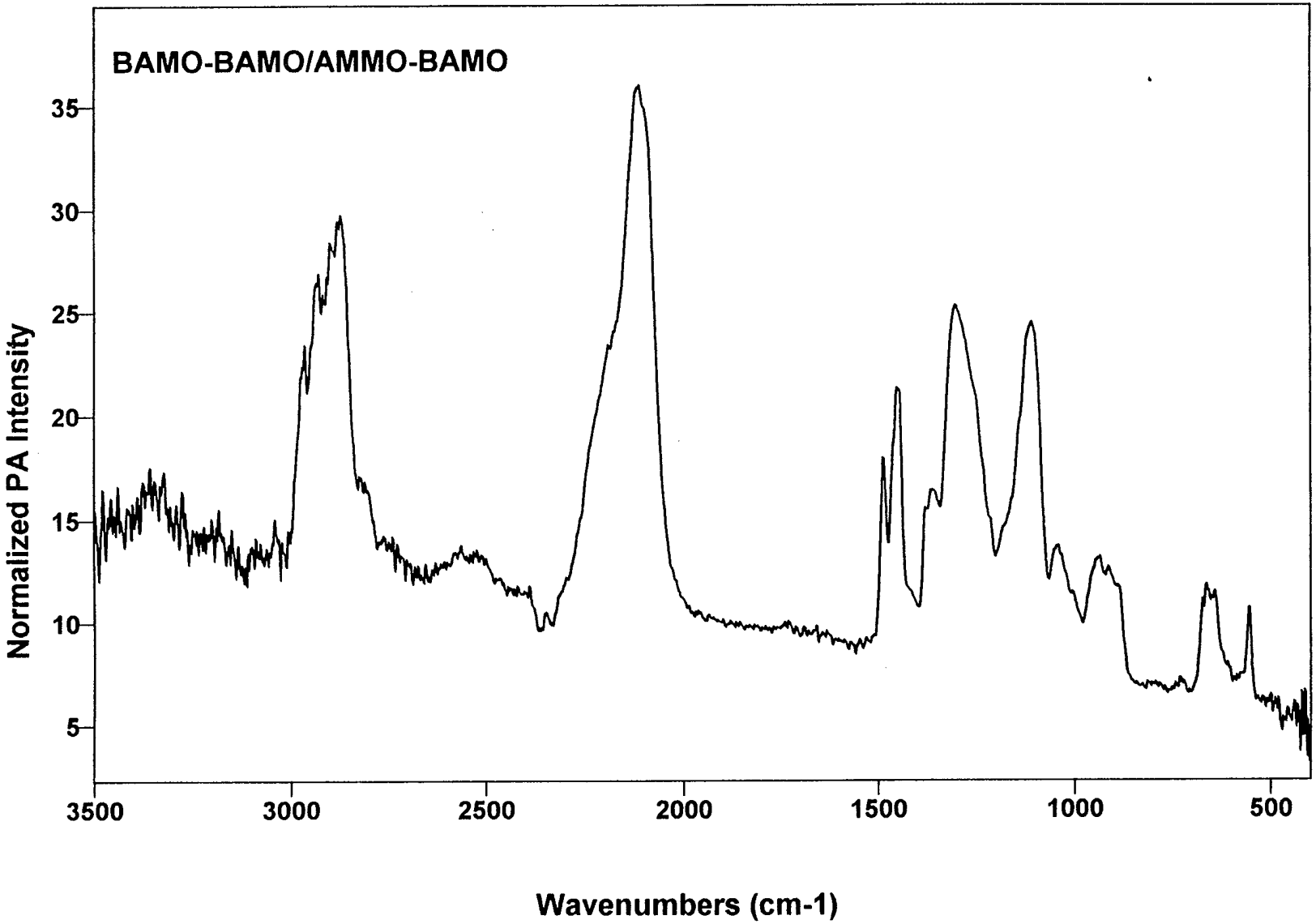
5
3500
3000
2500
2000
1500
1000
500
Wavenumbers (cm-1)


BEMO-BAMMO/THF
Normalized PA Intensity
80
70
60
50
40
30
20
10
4000
3500
3000
2500
2000
1500
1000
500
Wavenumbers (cm-1)


BAMO-BAMO/AMMO-BAMO
Normalized PA Intensity
35
30
25
20
15
10
5
3500
3000
2500
2000
1500
1000
500
Wavenumbers (cm-1)

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APPENDIX D:

SPECTRA OF PLASTICIZERS

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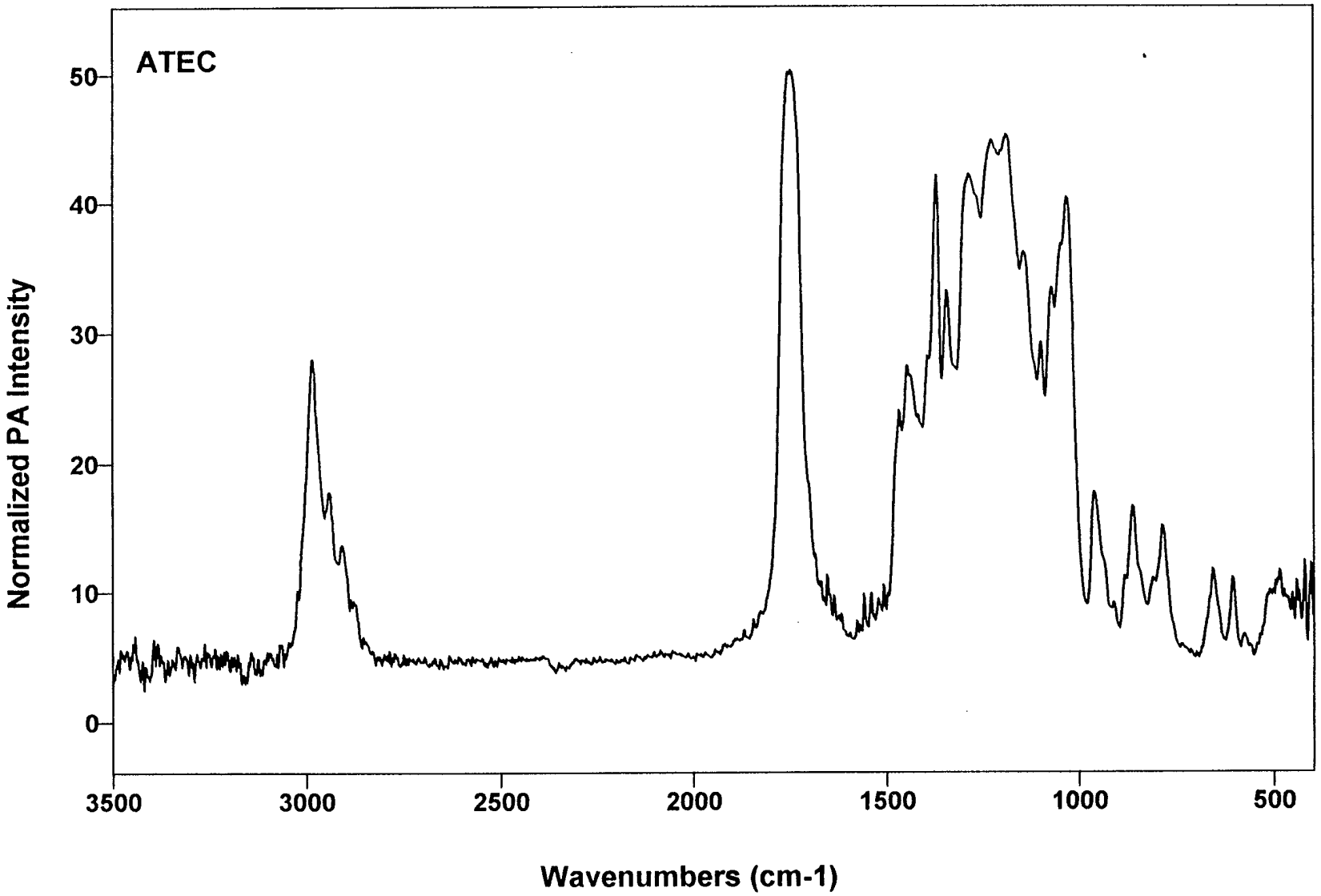
ATEC
Normalized PA Intensity
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40
30
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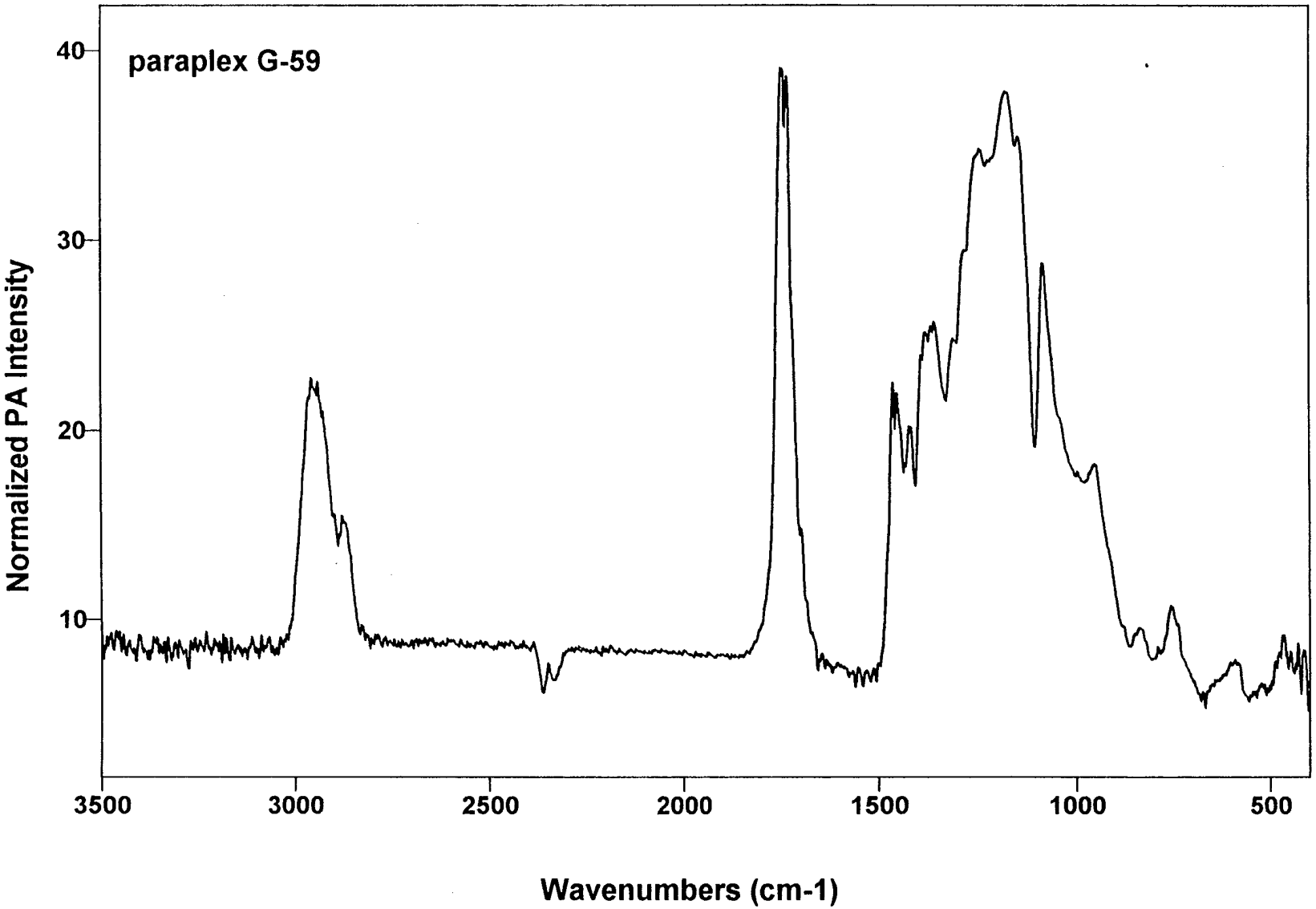
10
0
3500
3000
2500
2000
1500
1000
500
Wavenumbers (cm-1)


paraplex G-59
Normalized PA Intensity
40
30
20
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3500
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2500
2000
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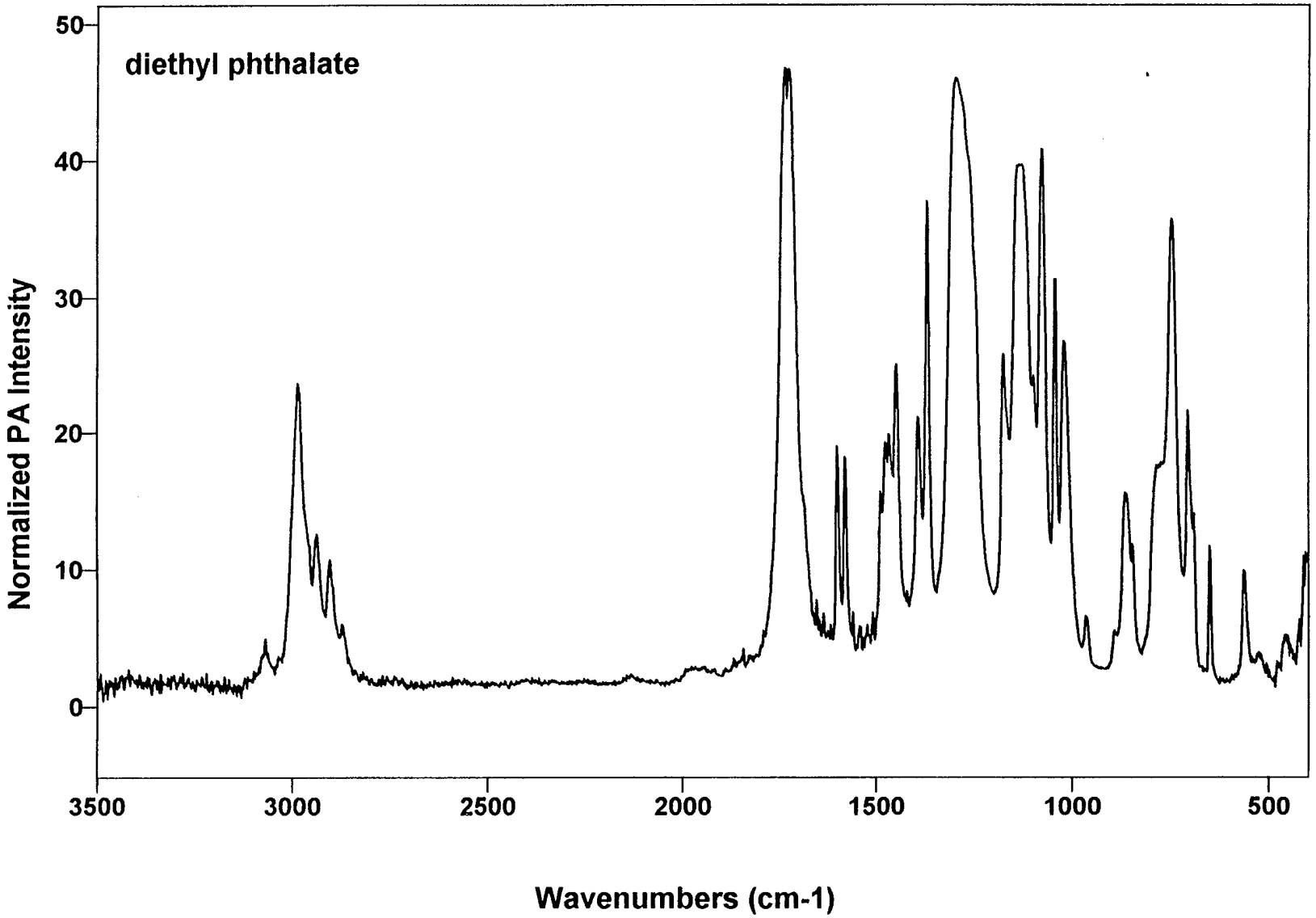
1000
500
Wavenumbers (cm-1)


diethyl phthalate
Normalized PA Intensity
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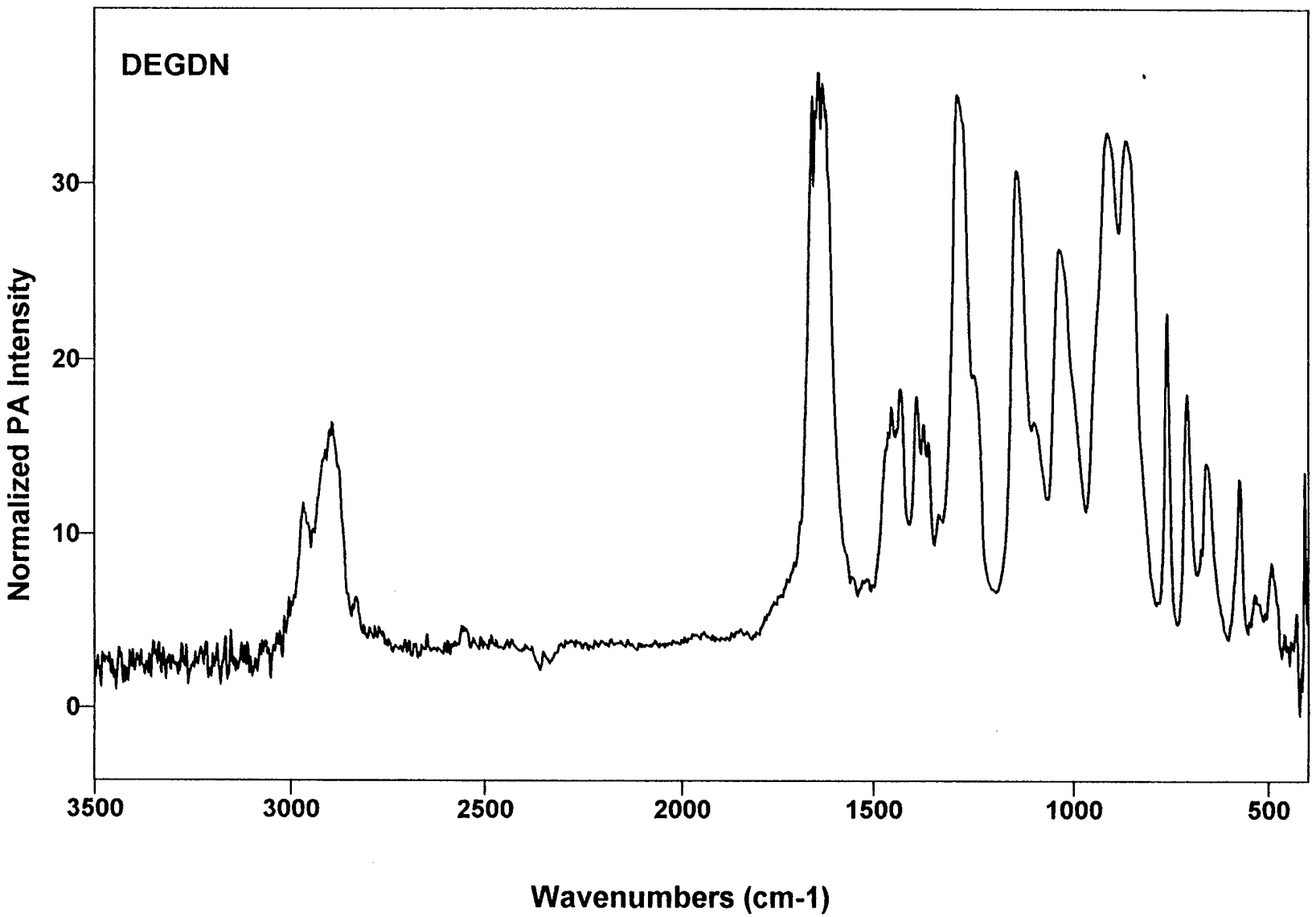
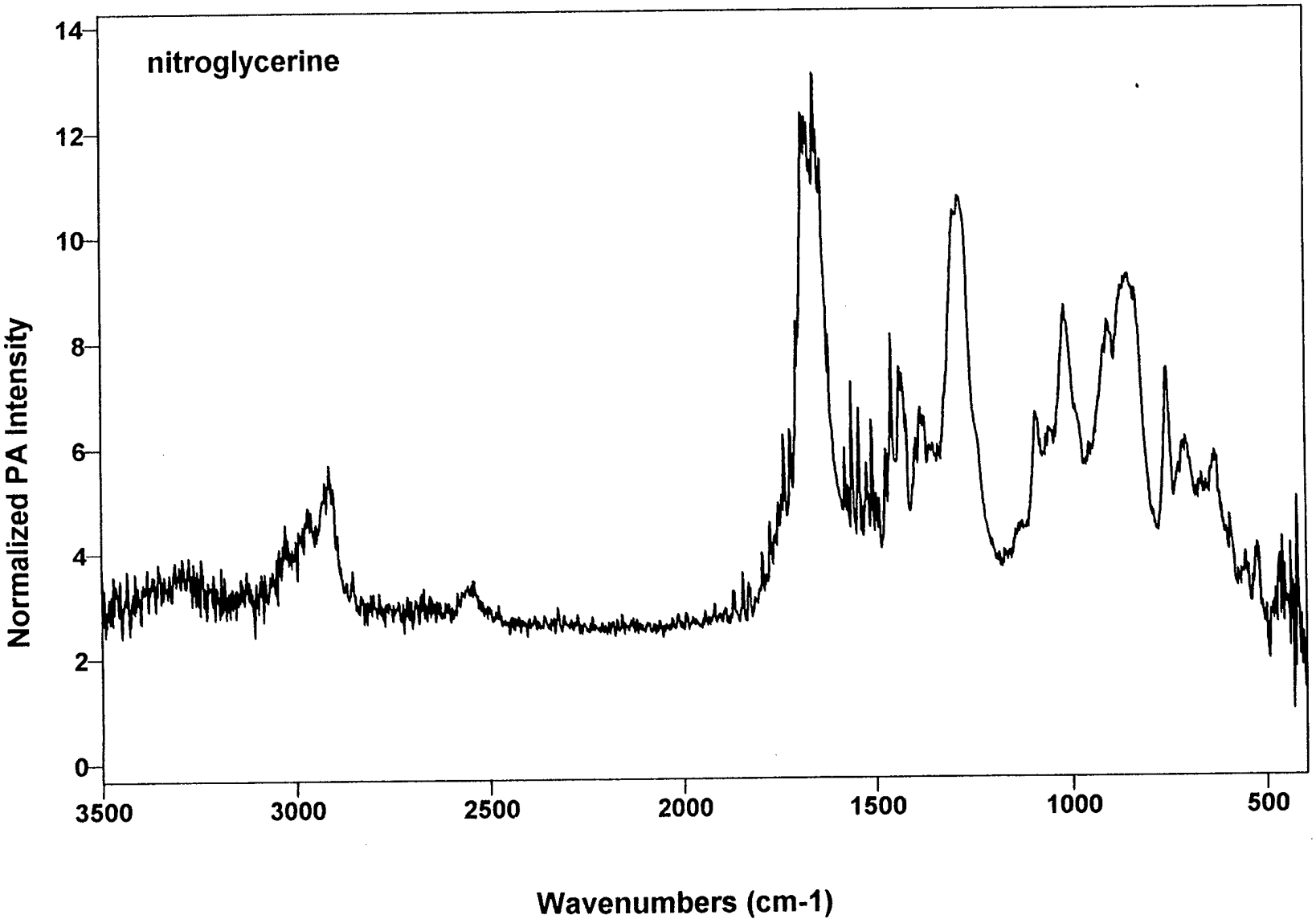
40
30
20
10
0
3500
3000
2500
2000
1500
1000
500
Wavenumbers (cm-1)


DEGDN
Normalized PA Intensity
30
20
10
0
3500
3000
2500
2000
1500
1000
500
Wavenumbers (cm-1)


nitroglycerine
Normalized PA Intensity
14
12
10
8
6
4
2
0
3500
3000
2500
2000
1500
1000
500
Wavenumbers (cm-1)

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APPENDIX E:

SPECTRA OF STABILIZERS

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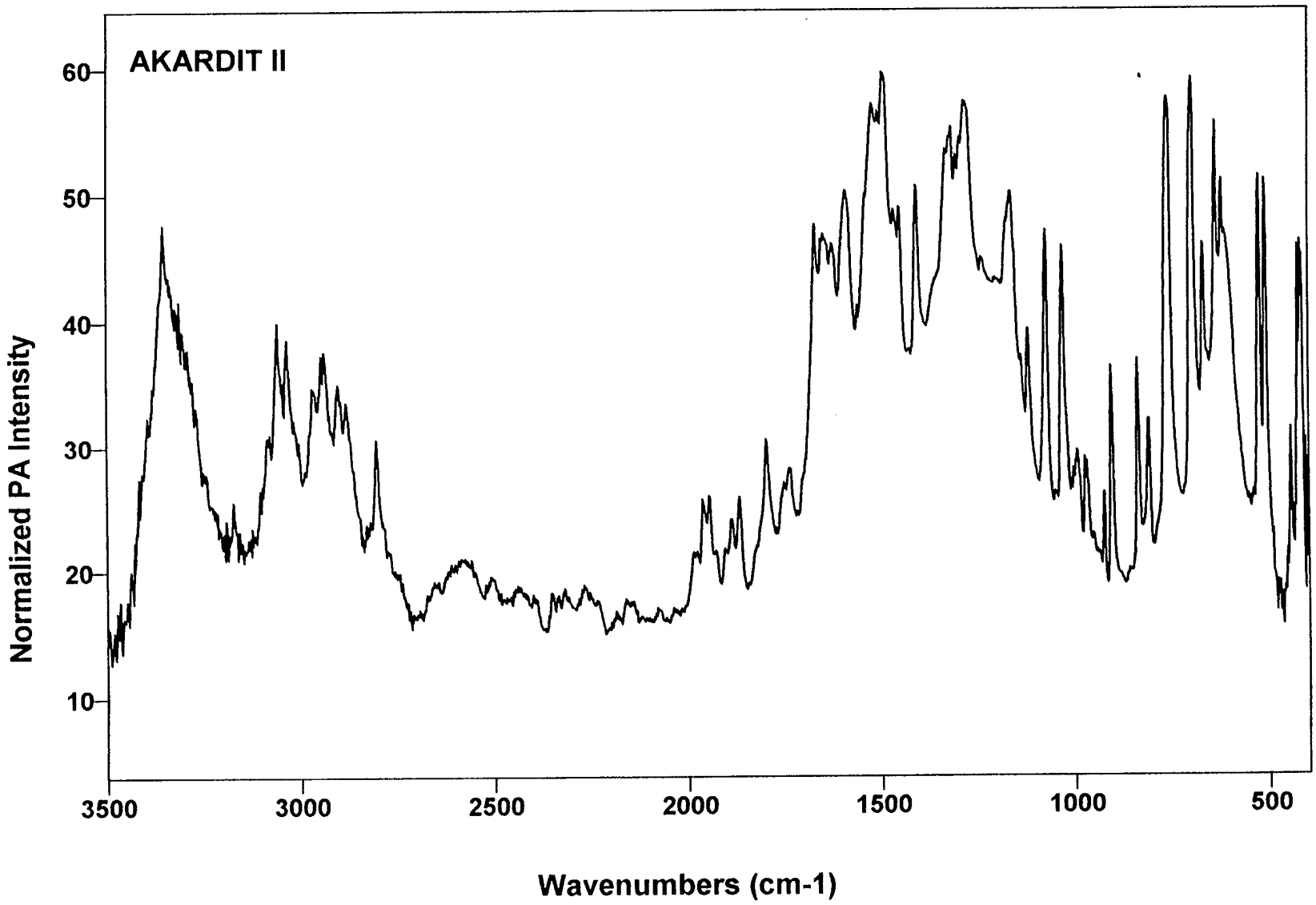
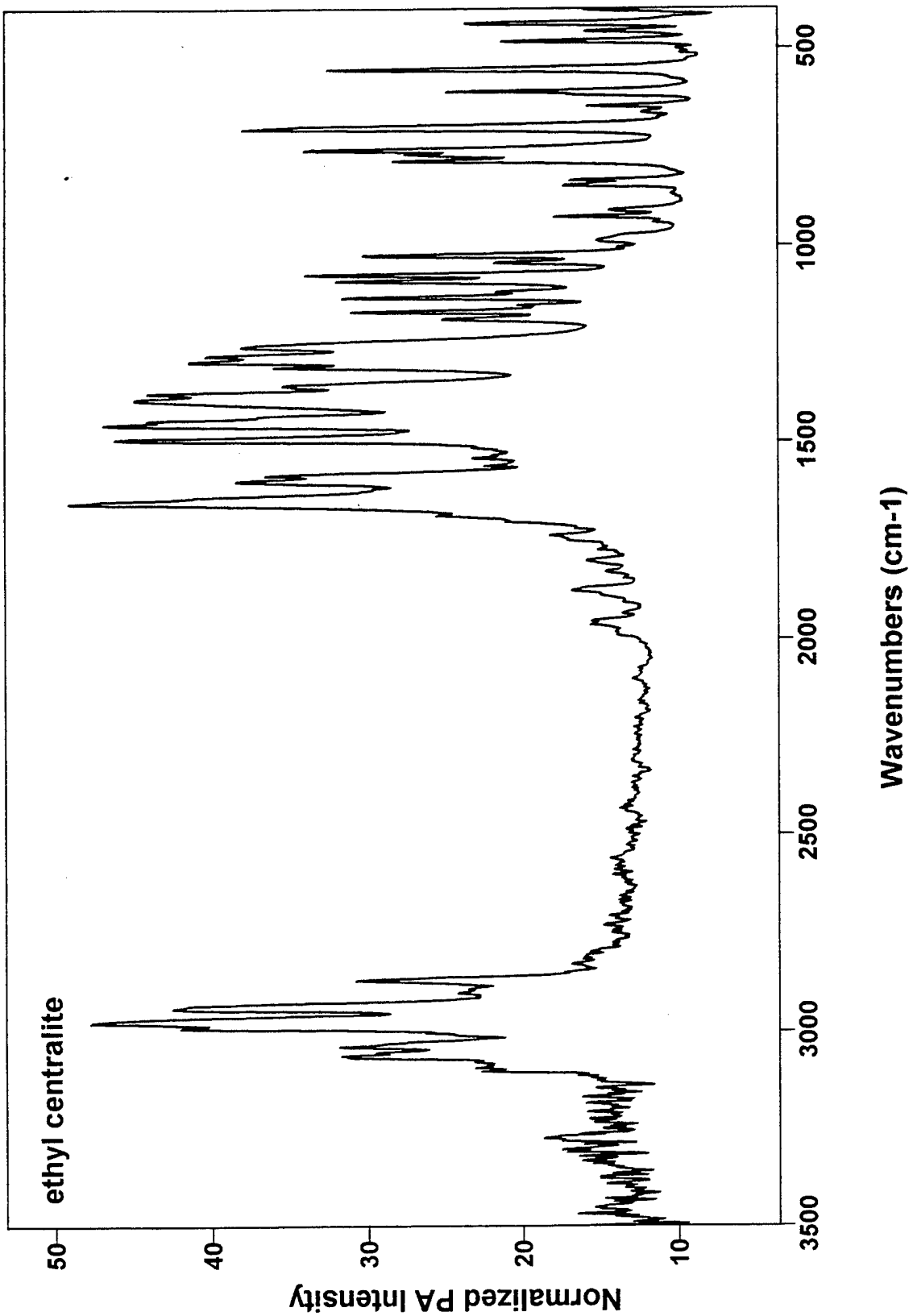
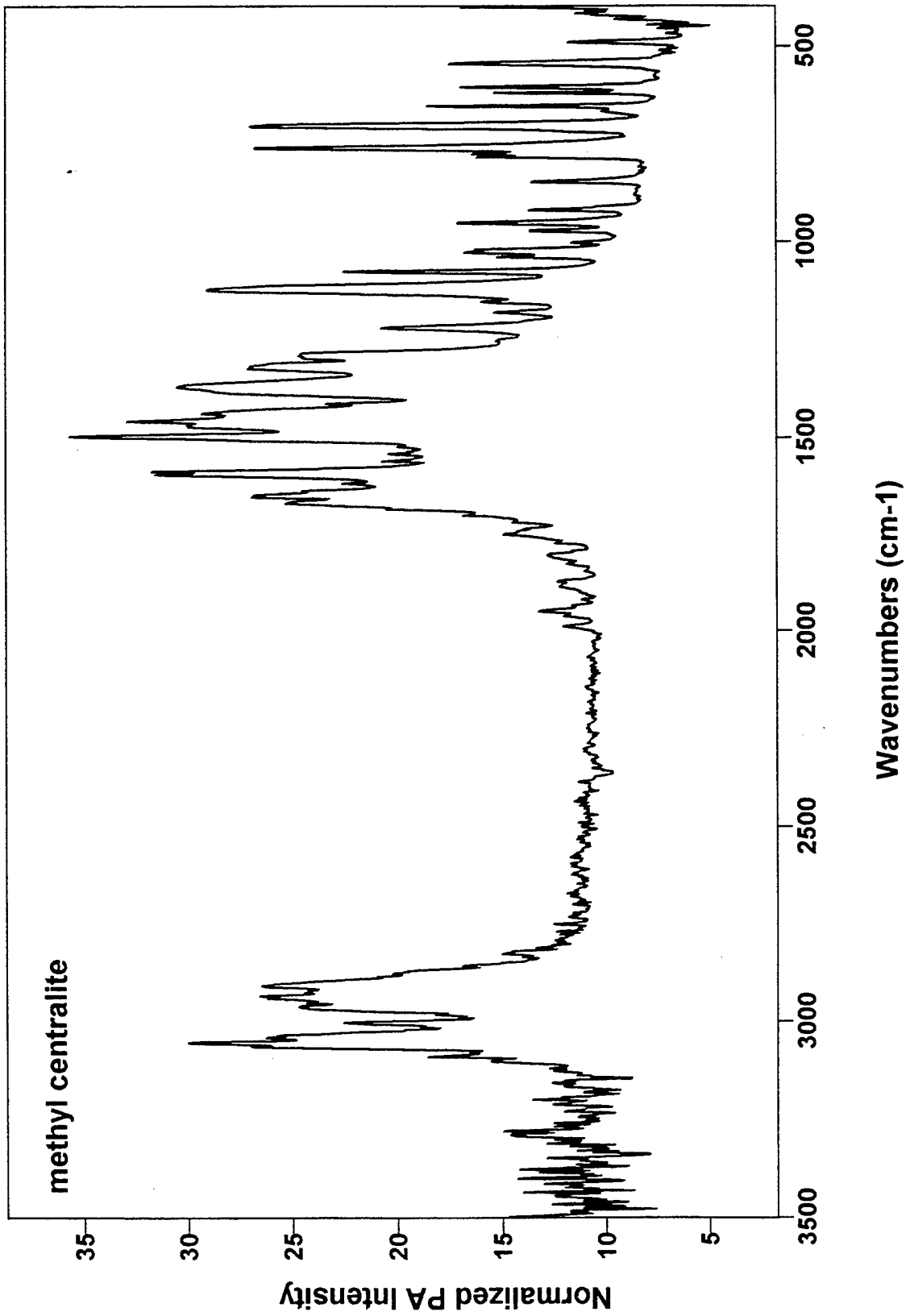

AKARDIT II
Normalized PA Intensity
60
50
40
30
20
10
3500
3000
2500
2000
1500
1000
500
Wavenumbers (cm-1)


ethyl centralite
Normalized PA Intensity
50
40
30
20
10
3500
3000
2500
2000
1500
1000
500
Wavenumbers (cm-1)


methyl centralite
Normalized PA Intensity
35
30
25
20
15
10
5
3500
3000
2500
2000
1500
1000
500
Wavenumbers (cm-1)

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APPENDIX F:

SPECTRA OF OTHERS

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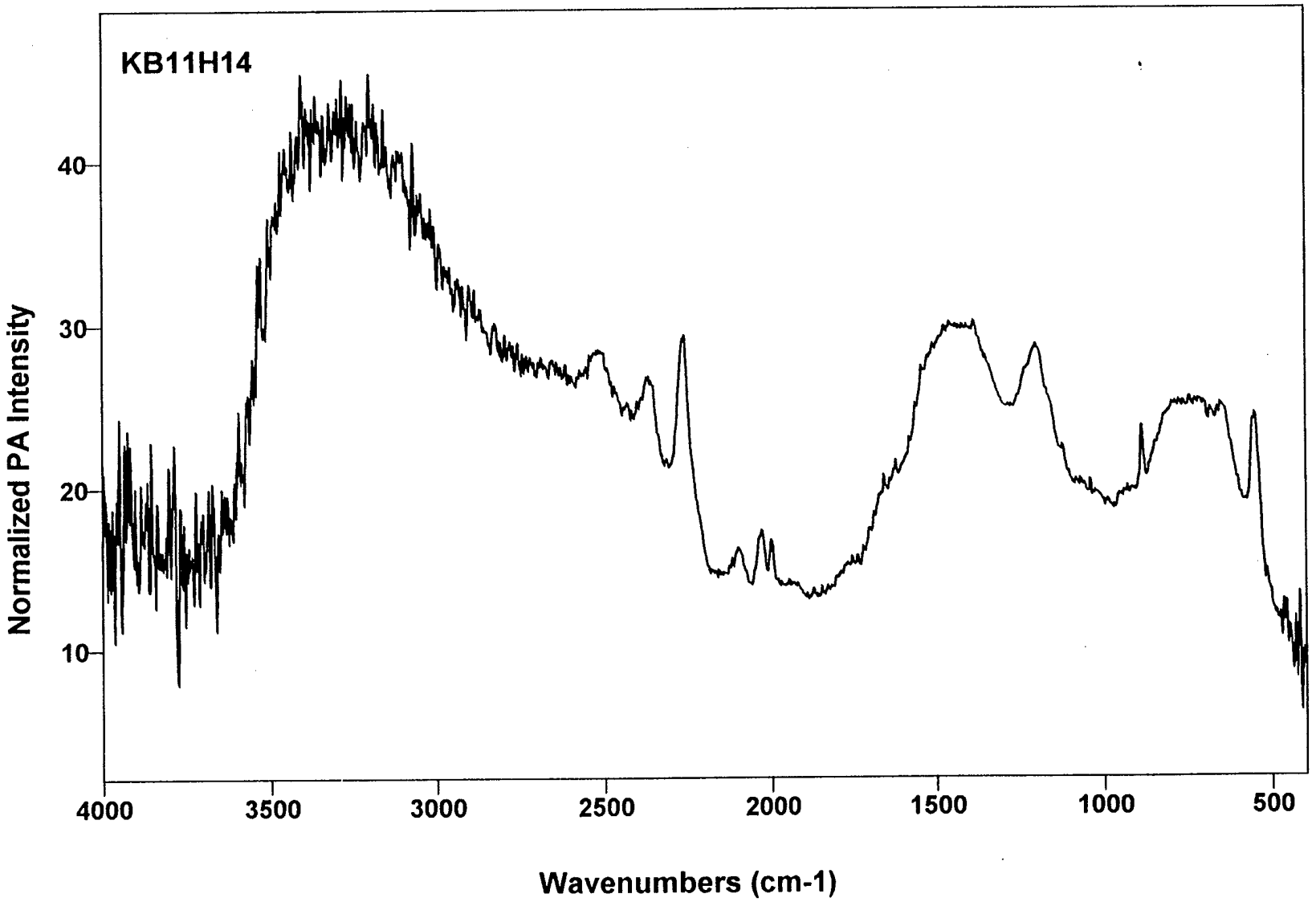
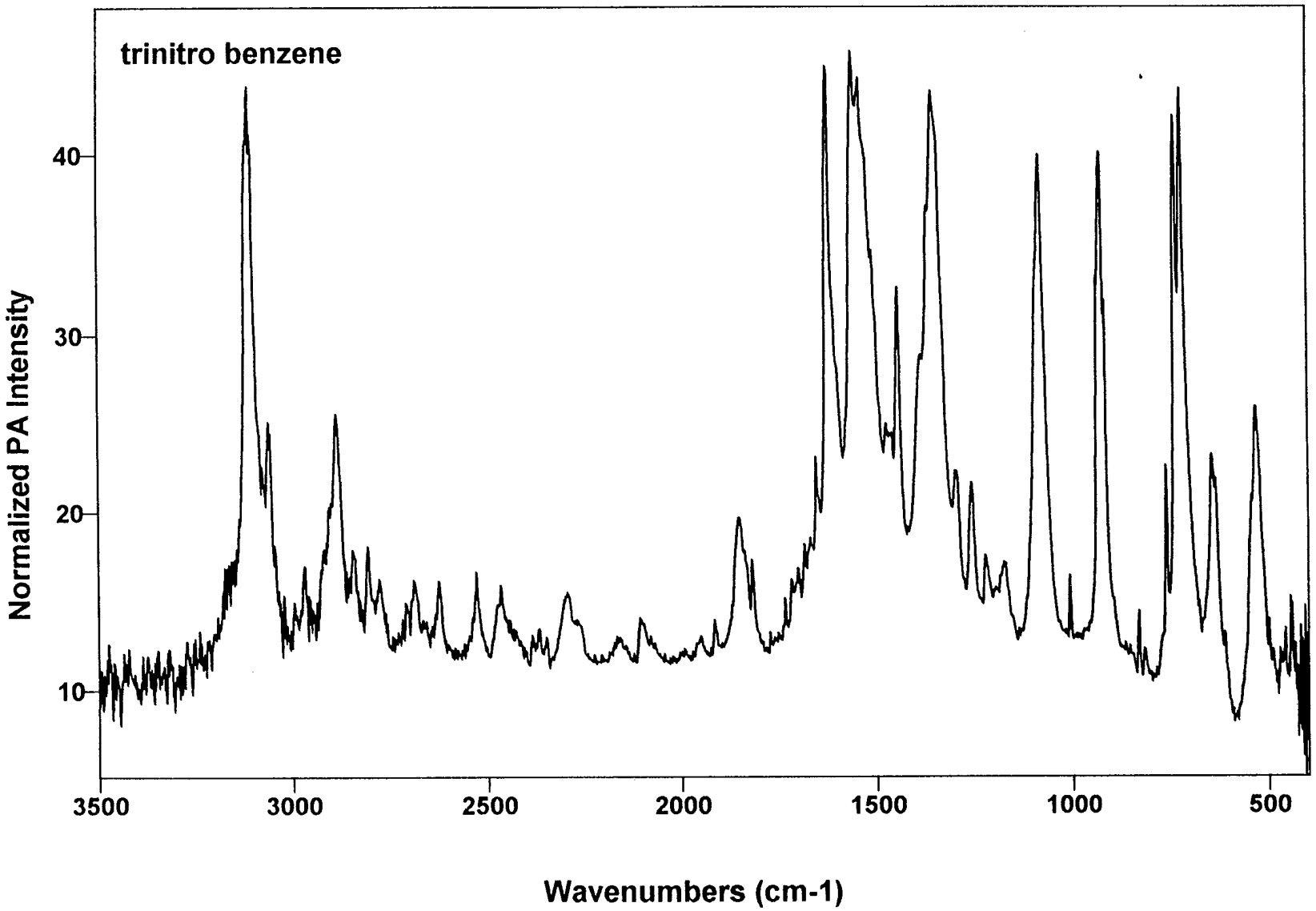
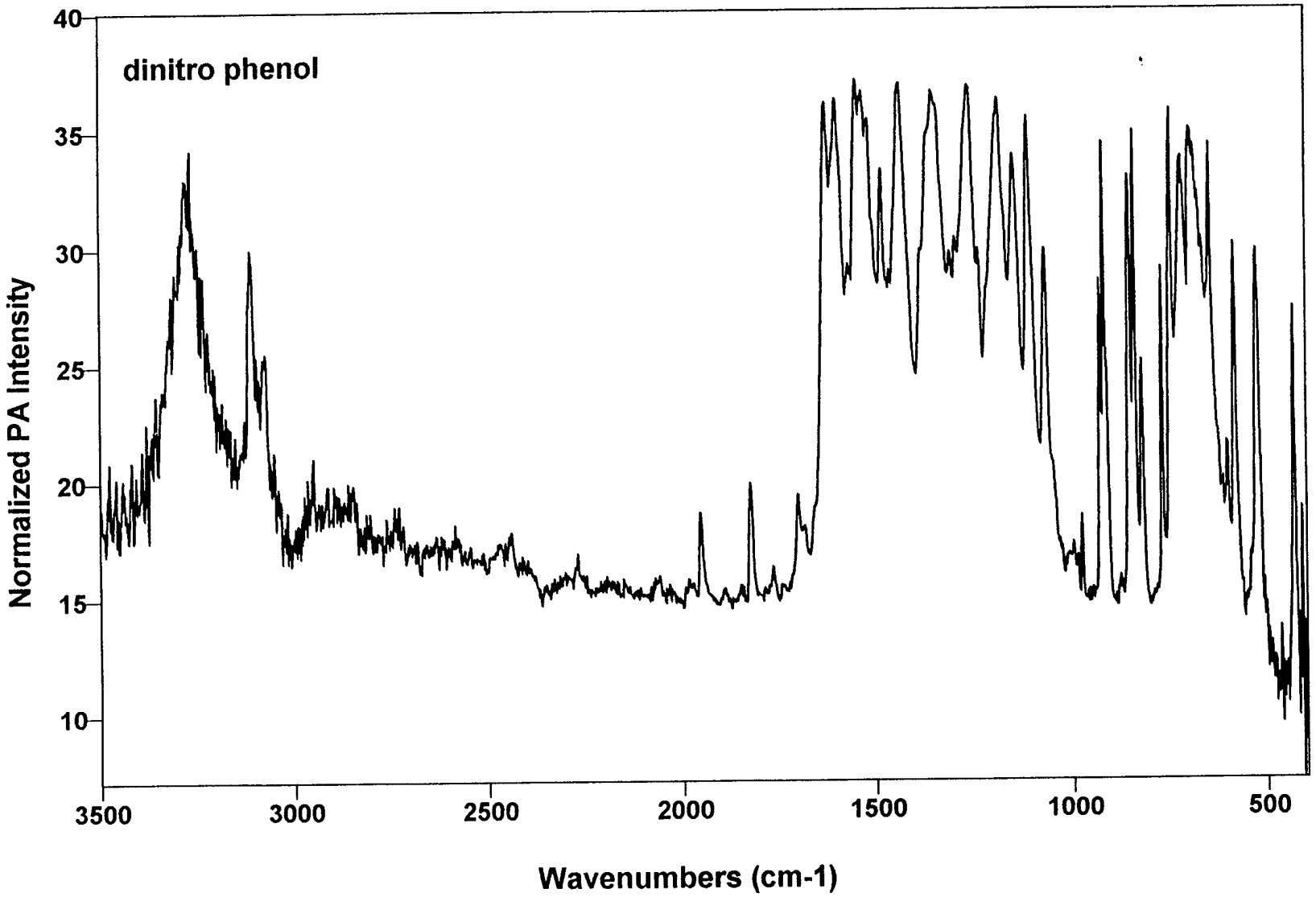

KB11H14
Normalized PA Intensity
40
30
20
10
4000
3500
3000
2500
2000
1500
1000
500
Wavenumbers (cm-1)


trinitro benzene
Normalized PA Intensity
40
30
20
10
3500
3000
2500
2000
1500
1000
500
Wavenumbers (cm-1)


dinitro phenol
Normalized PA Intensity
40
35
30
25
20
15
10
3500
3000
2500
2000
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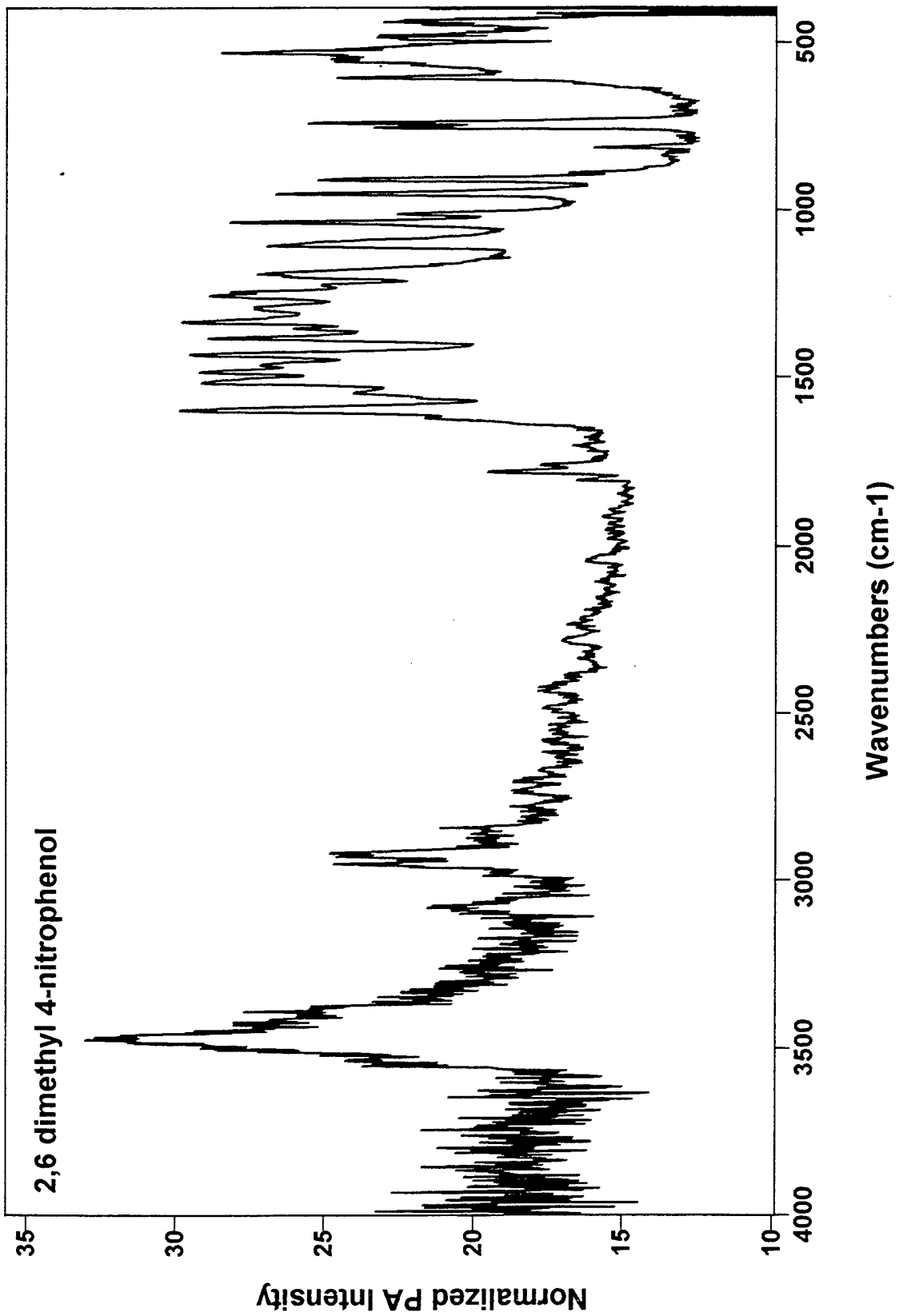
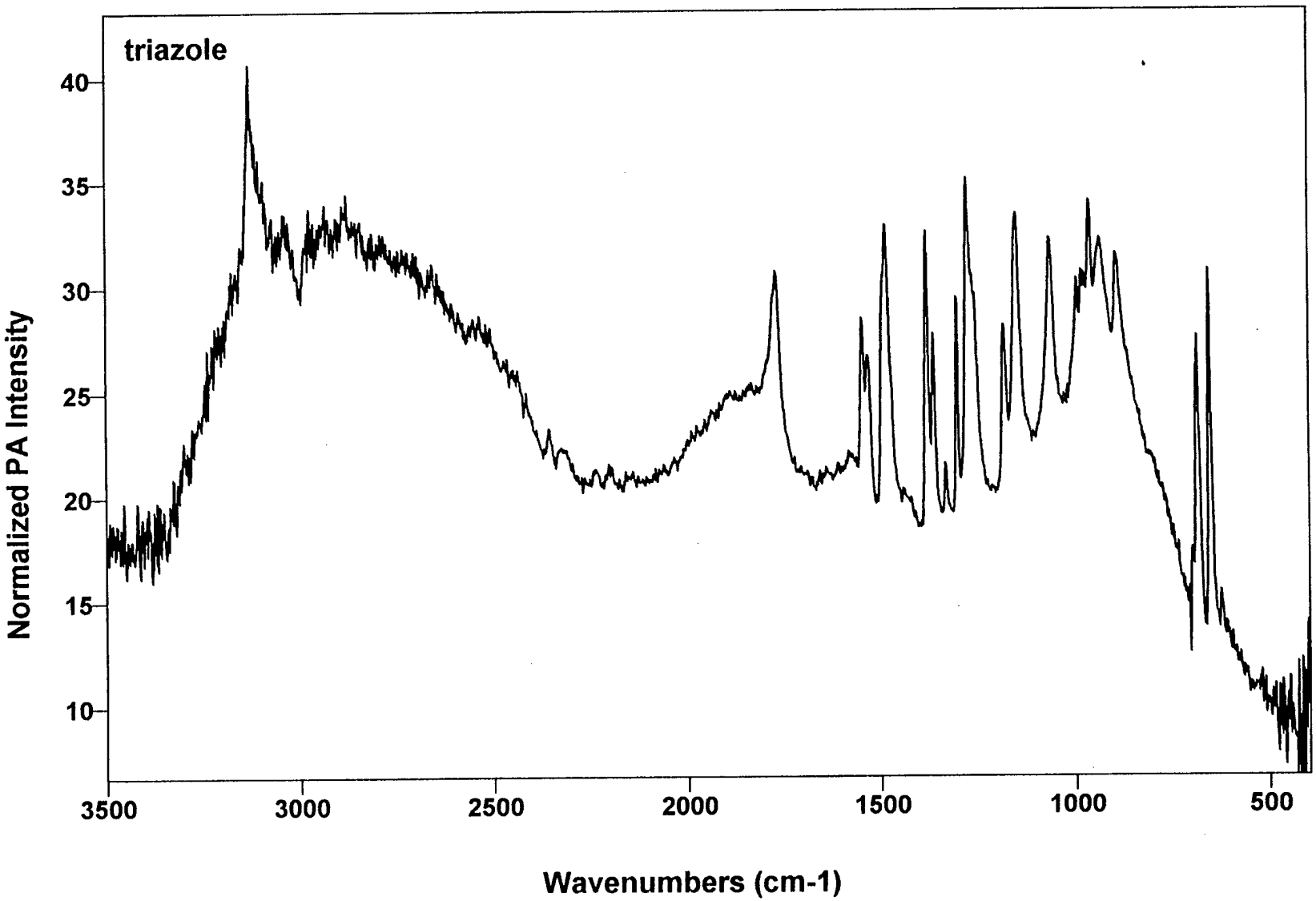
1000
500
Wavenumbers (cm-1)


2,6 dimethyl 4-nitrophenol
Normalized PA Intensity
Wavenumbers (cm-1)
35
30
25
20
15
10
4000
3500
3000
2500
2000
1500
1000
500


triazole
Normalized PA Intensity
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35
30
25
20
15
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3500
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2500
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1500
1000
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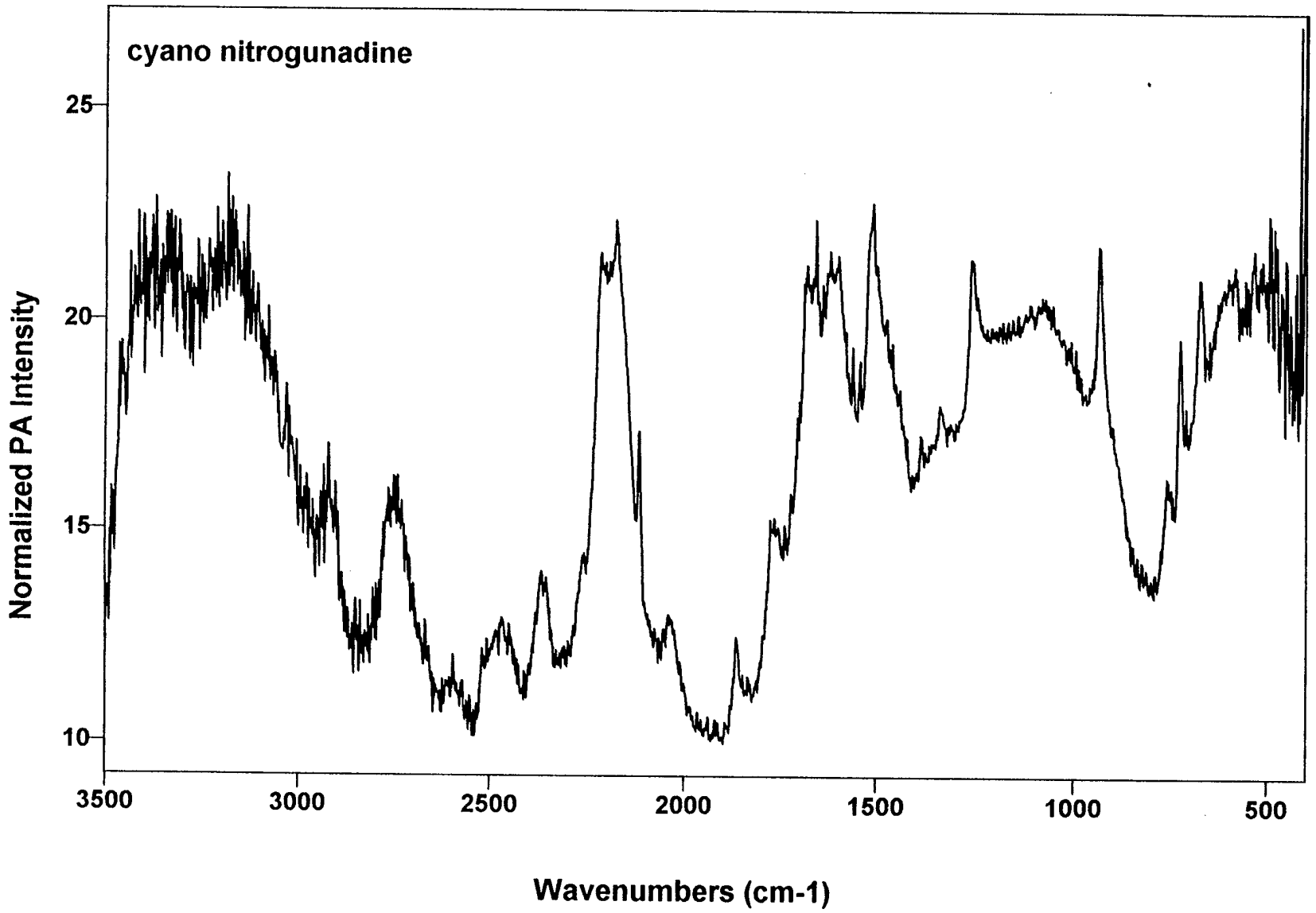
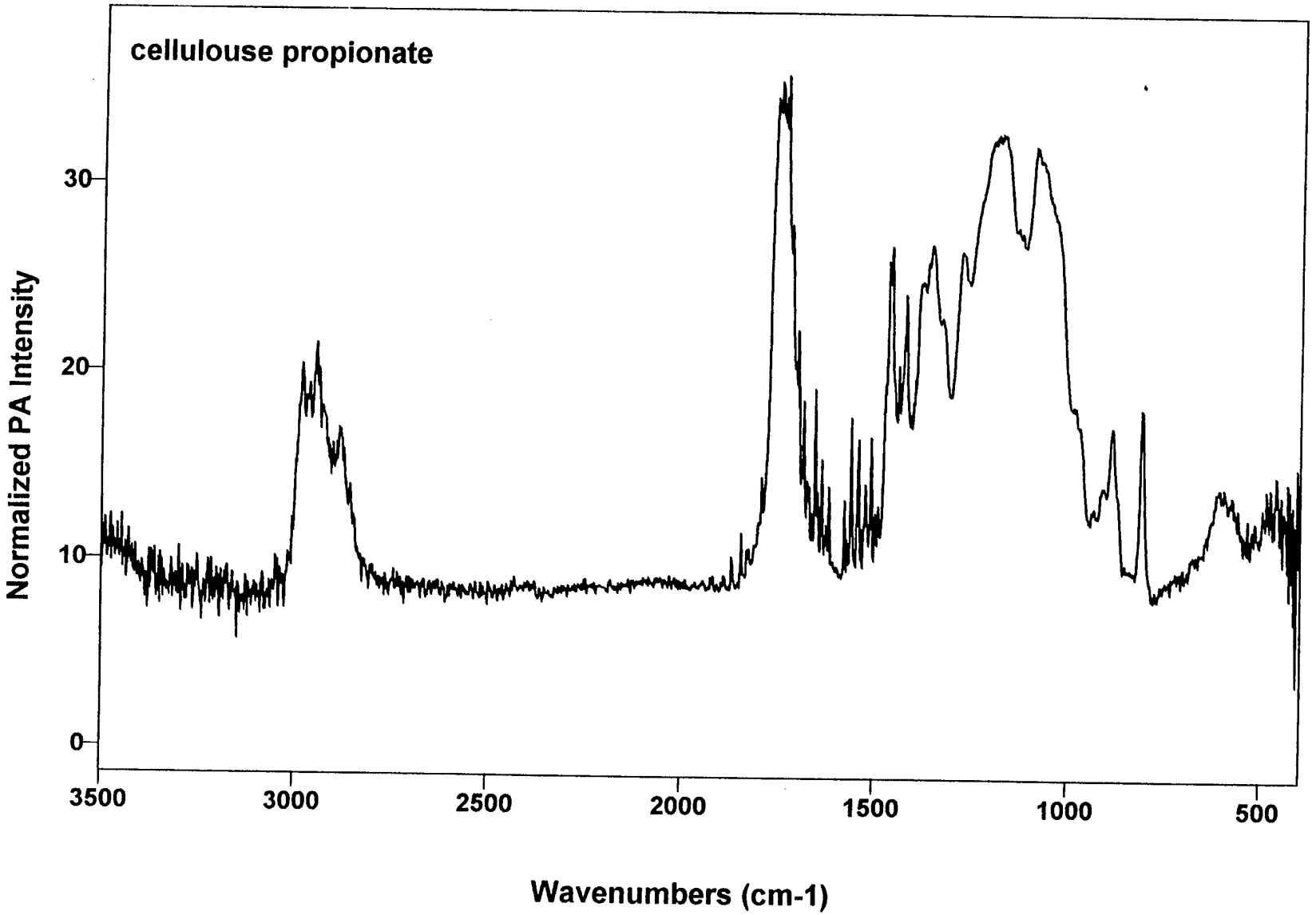
Wavenumbers (cm-1)


cyano nitrogunadine
Normalized PA Intensity
25
20
15
10
3500
3000
2500
2000
1500
1000
500
Wavenumbers (cm-1)


cellulouse propionate
Normalized PA Intensity
30
20
10
0
3500
3000
2500
2000
1500
1000
500
Wavenumbers (cm-1)

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LIST OF ABBREVIATIONS

AKARDIT II	-	Methyl Diphenyl Urea
AMMO	-	Azido Methyl Methyl Oxitane
ATEC	-	Acetyl Triethyl Citrate
ATR	-	Attenuated Total Reflectance
BAMO	-	Bis Azido Methyl Oxitane
BDNPA/F	-	Bis-Dinitro Propyl Acetal/Formal
BEMO	-	Bis Ethyl Methyl Oxitane
CAB	-	Cellulose Acetate Butyrate
CNNQ	-	Cyanonitroguanadine
CP	-	Cellulose Propionate
DANPE	-	Diazodo-Nitraza-Pentane
DEGDN	-	Diethylene Glycol Dinitrate
DMNP	-	2,6, Dimethyl 4-Nitrophenol
DNP	-	Dinitrophenol
DPA	-	Diphenyl Amine
EC	-	Ethyl Centralite
FTIR	-	Fourier Transform Infrared
HELP	-	High Energy Low Vulnerability Propellant
HMX	-	High Melting Explosive
IR	-	Infrared
JAG	-	JA2 with Paraplex G-59
JAX	-	JA2 with RDX
KS	-	Potassium Sulfate
MC	-	Methyl Centralite
NC	-	Nitrocellulose
NG	-	Nitroglycerine
NQ	-	Nitroguanidine
PA	-	Photoacoustic
PDNPA	-	Poly (Dinitro Propyl Acrylate)
RDX	-	Royal Developmental Explosive
TNAZ	-	1,3,3-Trinitroazetidine
TNB	-	Trinitobenzene
TNT	-	Trinitrotoluene
TPE	-	Thermoplastic Elastomer

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